```
=> dhis
DHIS IS NOT A RECOGNIZED COMMAND
The previous command name entered was not recognized by the system.
For a list of commands available to you in the current file, enter
"HELP COMMANDS" at an arrow prompt (=>).
=> d his
     (FILE 'HOME' ENTERED AT 11:48:54 ON 29 AUG 2007)
     FILE 'STNGUIDE' ENTERED AT 11:49:06 ON 29 AUG 2007
     FILE 'HCAPLUS' ENTERED AT 11:50:33 ON 29 AUG 2007
Ll
              O SEA ABB=ON PLU=ON "CYCLOHEXANEDIACETIC ACID"+PFT, OLD, NEW/CT
     FILE 'REGISTRY' ENTERED AT 11:55:02 ON 29 AUG 2007
                E CYCLOHEXANEDIACETIC/CN
     FILE 'HCAPLUS' ENTERED AT 11:55:03 ON 29 AUG 2007
                S E4
     FILE 'REGISTRY' ENTERED AT 11:56:31 ON 29 AUG 2007
L2
              1 S E4/CN
     FILE 'HCAPLUS' ENTERED AT 11:56:31 ON 29 AUG 2007
L3
              8 S L2
                E CYCLOHEXANEDIACETIC ACID ANHYDRIDE
         230564 S ANHYDRIDE
              0 S L4 (3W) L3
L5
L6
         230564 SEA ABB=ON PLU=ON ANHYDRIDE
L7
          1633 S ABB=ON PLU=ON MONOAMIDE
         19338 S ABB=ON PLU=ON PRECIPITATION+PFT, OLD, NEW/CT
L8
              2 S ABB=ON PLU=ON ACIDIFICATION+PFT, NEW, OLD/CT
L9
         214418 S ABB=ON PLU=ON AMMONIA
L10
              O S "HYDROCHLORIC ACID"+PFT, OLD, NEW/CT
L11
                E HYDROCHLORIC ACID
L12
             12 S E5
L13
              O S ABB=ON PLU=ON "ACETIC ANHDRIDE"+PFT, NEW, OLD/CT
                E ACETIC ANHYDRIDE
         247054 S ACETIC
L14
L15
          26509 S L14 (1W) L4
        6805049 S ABB=ON PLU=ON SYNTH OR SYNTHE? OR PREPARTION OR PRODUC? OR PR
L16
         157279 S ABB=ON PLU=ON AMINATION+PFT, OLD, NEW, RT/CT
L17
                E US4024175/PN
              1 S E3
L18
                SELECT RN L18 1
     FILE 'REGISTRY' ENTERED AT 12:26:48 ON 29 AUG 2007
L19
             11 S E1-E11
              O S ABB=ON PLU=ON "1,1-CYCLOHEXANEDIACETIC ACID"+RTCS, NEW, OLD/C
L20
L21
              O S ABB=ON PLU=ON GABAPENTINE+RTCS, NEW, OLD, PFT/CT
L22
              7 S GABAPENTIN
L23
              0 S L22 AND L2
L24
              0 S L22 AND L3
     FILE 'HCAPLUS' ENTERED AT 12:40:47 ON 29 AUG 2007
L25
          1956 S GABAPENTIN
```

```
L26
            0 S L25 AND L3
L27
            20 S L25 AND L6
L28
            4 S L27 AND L7
           99 S 1,1-CYCLOHEXANEDIACETIC ACID
L29
L30
            4 S L29 (3W) L6
L31
            3 S L30 NOT L28
L32
            2 S L29 AND L17 NOT L28
L33
            2 S L29 AND L8
            0 S L29 AND L9 NOT L28
L34
   FILE 'CASREACT' ENTERED AT 12:53:00 ON 29 AUG 2007
L35
          26 S 1,1-CYCLOHEXANEDIACETIC ACID
        19903 S AMINATION
L36
L37
          235 S PRECIPITATION
L38
          243 S MONOAMIDE
           1 S 1,1-CYCLOHEXANEDIACETIC ACID ANHYDRIDE
L39
            2 S L35 AND L36
L40
         3188 S ACIDIFICATION
L41
L42
           6 S L38 AND L35
L43
            2 S L42 AND L37
L44
         8354 S AMMONIA
           3 S L44 AND L29
L45
            1 S L45 NOT L28
L46
```

# => log off

ALL L# QUERIES AND ANSWER SETS ARE DELETED AT LOGOFF LOGOFF? (Y)/N/HOLD:y

STN INTERNATIONAL LOGOFF AT 13:07:03 ON 29 AUG 2007

# Connecting via Winsock to STN

NEWS IPC8

```
Welcome to STN International! Enter x:x
LOGINID: SSPTAYKC1621
PASSWORD:
TERMINAL (ENTER 1, 2, 3, OR ?):2
 * * * * * * * *
                     Welcome to STN International
NEWS 1
                 Web Page for STN Seminar Schedule - N. America
                 New CAS web site launched
 NEWS 2 MAY 01
                 CA/CAplus Indian patent publication number format defined
 NEWS 3
         MAY 08
 NEWS 4 MAY 14
                 RDISCLOSURE on STN Easy enhanced with new search and display
                  fields
 NEWS 5 MAY 21
                 BIOSIS reloaded and enhanced with archival data
 NEWS 6 MAY 21
                 TOXCENTER enhanced with BIOSIS reload
                 CA/CAplus enhanced with additional kind codes for German
 NEWS 7 MAY 21
                 patents .
 NEWS 8 MAY 22
                 CA/CAplus enhanced with IPC reclassification in Japanese
                 patents
                 CA/CAplus enhanced with pre-1967 CAS Registry Numbers
         JUN 27
 NEWS 9
 NEWS 10 JUN 29
                 STN Viewer now available
                 STN Express, Version 8.2, now available
 NEWS 11
         JUN 29
 NEWS 12
         JUL 02
                 LEMBASE coverage updated
         JUL 02
                 LMEDLINE coverage updated
 NEWS 13
                 SCISEARCH enhanced with complete author names
 NEWS 14 JUL 02
 NEWS 15 JUL 02
                 CHEMCATS accession numbers revised
 NEWS 16 JUL 02
                 CA/CAplus enhanced with utility model patents from China
                 CAplus enhanced with French and German abstracts
 NEWS 17 JUL 16
 NEWS 18 JUL 18
                  CA/CAplus patent coverage enhanced
                 USPATFULL/USPAT2 enhanced with IPC reclassification
 NEWS 19 JUL 26
 NEWS 20 JUL 30 USGENE now available on STN
                 CAS REGISTRY enhanced with new experimental property tags
 NEWS 21 AUG 06
 NEWS 22 AUG 06
                  BEILSTEIN updated with new compounds
                  FSTA enhanced with new thesaurus edition
 NEWS 23 AUG 06
                  CA/CAplus enhanced with additional kind codes for granted
 NEWS 24 AUG 13
                  patents
                  CA/CAplus enhanced with CAS indexing in pre-1907 records
 NEWS 25 AUG 20
                  Full-text patent databases enhanced with predefined
 NEWS 26 AUG 27
                  patent family display formats from INPADOCDB
                  USPATOLD now available on STN
 NEWS 27
          AUG 27
                  CAS REGISTRY enhanced with additional experimental
 NEWS 28
          AUG 28
                  spectral property data
               29 JUNE 2007: CURRENT WINDOWS VERSION IS V8.2,
 NEWS EXPRESS
               CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP),
               AND CURRENT DISCOVER FILE IS DATED 05 JULY 2007.
               STN Operating Hours Plus Help Desk Availability
 NEWS HOURS
 NEWS LOGIN
               Welcome Banner and News Items
```

For general information regarding STN implementation of IPC 8

Enter NEWS followed by the item number or name to see news on that specific topic.

All use of STN is subject to the provisions of the STN Customer agreement. Please note that this agreement limits use to scientific research. Use for software development or design or implementation of commercial gateways or other similar uses is prohibited and may result in loss of user privileges and other penalties.

FILE 'HOME' ENTERED AT 11:48:54 ON 29 AUG 2007

=> file stnguide
COST IN U.S. DOLLARS

FULL ESTIMATED COST

SINCE FILE TOTAL ENTRY SESSION 0.21 0.21

FILE 'STAGUIDE' ENTERED AT 11.4

FILE 'STNGUIDE' ENTERED AT 11:49:06 ON 29 AUG 2007 USE IS SUBJECT TO THE TERMS OF YOUR CUSTOMER AGREEMENT COPYRIGHT (C) 2007 AMERICAN CHEMICAL SOCIETY (ACS)

FILE CONTAINS CURRENT INFORMATION.
LAST RELOADED: Aug 24, 2007 (20070824/UP).

=> file hcaplus
COST IN U.S. DOLLARS

SINCE FILE TOTAL ENTRY SESSION 0.12 0.33

FULL ESTIMATED COST

FILE 'HCAPLUS' ENTERED AT 11:50:33 ON 29 AUG 2007 USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT. PLEASE SEE "HELP USAGETERMS" FOR DETAILS.
COPYRIGHT (C) 2007 AMERICAN CHEMICAL SOCIETY (ACS)

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FILE COVERS 1907 - 29 Aug 2007 VOL 147 ISS 10 FILE LAST UPDATED: 28 Aug 2007 (20070828/ED)

New CAS Information Use Policies, enter HELP USAGETERMS for details.

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> sea abb=on plu=on "cyclohexanediacetic acid"=pft,old,new/ct NUMERIC VALUE NOT VALID 'PFT,OLD,NEW' Numeric values may contain 1-8 significant figures. If range notation is used, both the beginning and the end of the range must be specified, e.g., '250-300/MW'. Expressions such as '250-/MW' are not

allowed. To search for values above or below a given number, use the >, =>, <, or <= operators, e.g., 'MW => 250'. Text terms cannot be used in numeric expressions. If you specify a unit, it must be dimensionally correct for that field code. To see the unit designations for field codes in the current file, enter "DISPLAY UNIT ALL" at an arrow prompt (=>).

=> display unit all UNITS CONVERSION IS NOT AVAILABLE IN THE CURRENT FILE

=> sea abb=on plu=on "cyclohexanediacetic acid"+pft, old, new/ct
'"CYCLOHEXANEDIACETIC ACID"' NOT IN RELATIONSHIP FILE
RELATIONSHIP CODE ',OLD,NEW' IGNORED
L1 0 "CYCLOHEXANEDIACETIC ACID"+PFT, OLD, NEW/CT (1 TERM)

=> e cyclohexanediacetic/cn
 REG1stRY INITIATED

Substance data EXPAND from CAS REGISTRY in progress...

E1	1	CYCLOHEXANEDECYLAMINE, (AMINOMETHYL)-4-BUTYL-/CN
E2	1	CYCLOHEXANEDECYLAMINE, HYDROCHLORIDE/CN
E3	0>	CYCLOHEXANEDIACETIC/CN
E4	1	CYCLOHEXANEDIACETIC ACID/CN
E5	1	CYCLOHEXANEDIACRYLATE/CN
E6	1 '	CYCLOHEXANEDIAMINE/CN
E7	1	CYCLOHEXANEDIAMINE, C,C,C,C-TETRAMETHYL-/CN
E8	1	CYCLOHEXANEDIAMINE, C-((AMINOCYCLOHEXYL)METHYL)-/CN
E9	1	CYCLOHEXANEDIAMINE, C-((AMINOCYCLOHEXYL)METHYL)-C-METHYL-/CN
E10	1	CYCLOHEXANEDIAMINE, HOMOPOLYMER/CN
E11	1	CYCLOHEXANEDIAMINE, METHYL-/CN
E12	1	CYCLOHEXANEDIAMINE, N,N'-DIPHENYL-/CN

# => s e4

REG1stRY INITIATED

Substance data SEARCH and crossover from CAS REGISTRY in progress... Use DISPLAY HITSTR (or FHITSTR) to directly view retrieved structures.

L3 8 L2

=> display hitstr ENTER (L3), L# OR ?:13 ENTER ANSWER NUMBER OR RANGE (1):1-8

- L3 ANSWER 1 OF 8 HCAPLUS COPYRIGHT 2007 ACS on STN
- IT 152848-09-4, Cyclohexanediacetic acid
   RL: CPS (Chemical process); PEP (Physical, engineering or chemical
   process); TEM (Technical or engineered material use); PROC (Process); USES
   (Uses)

(heat-transfer medium composition with corrosion prevention in pipings of

water cooling system)

152848-09-4 HCAPLUS

Cyclohexanediacetic acid (9CI) (CA INDEX NAME) CN



RN

ANSWER 2 OF 8 HCAPLUS COPYRIGHT 2007 ACS on STN L3

152848-09-4, Cyclohexanediacetic acid IT

RL: NUU (Other use, unclassified); USES (Uses)

(cleaning of filtration membranes using peracids)

152848-09-4 HCAPLUS RN

Cyclohexanediacetic acid (9CI) (CA INDEX NAME) CN



ANSWER 3 OF 8 HCAPLUS COPYRIGHT 2007 ACS on STN L3

152848-09-4, Cyclohexanediacetic acid IT

RL: ARU (Analytical role, unclassified); ANST (Analytical study)

(baroresistant buffer mixts. for biochem. analyses)

152848-09-4 HCAPLUS RN

Cyclohexanediacetic acid (9CI) (CA INDEX NAME) CN



ANSWER 4 OF 8 HCAPLUS COPYRIGHT 2007 ACS on STN L3

152848-09-4, Cyclohexanediacetic acid

RL: RCT (Reactant); RACT (Reactant or reagent)

(microwave irradiation process for preparing  $\ensuremath{\mathsf{Me}}$  carboxylate esters from carboxylate salts or carboxylic acids and di-Me carbonate)

152848-09-4 HCAPLUS RN

Cyclohexanediacetic acid (9CI) (CA INDEX NAME) CN



ANSWER 5 OF 8 HCAPLUS COPYRIGHT 2007 ACS on STN L3

152848-09-4D, Cyclohexanediacetic acid, acrylic-alkyd polymers IT RL: TEM (Technical or engineered material use); USES (Uses) (alkyd/acrylic latexes for cleaning, polishing, and protecting hard surfaces)

152848-09-4 HCAPLUS RN

Cyclohexanediacetic acid (9CI) (CA INDEX NAME) CN



L3

ANSWER 6 OF 8 HCAPLUS COPYRIGHT 2007 ACS on STN 152848-09-4D, Cyclohexanediacetic acid, anhydrides

RL: RCT (Reactant); RACT (Reactant or reagent)

(aqueous process for preparing amido-carboxylic acids by amidation of an amino

acid with a carboxylic acid anhydride)

152848-09-4 HCAPLUS RN

Cyclohexanediacetic acid (9CI) (CA INDEX NAME) CN



L3 ANSWER 7 OF 8 HCAPLUS COPYRIGHT 2007 ACS on STN

152848-09-4, Cyclohexanediacetic acid IT

RL: RCT (Reactant); RACT (Reactant or reagent)

(preparation of amido-carboxylic acids from lactams/amino acids and carboxylic acids/esters wherein hydrolysis and amidation reactions are conducted simultaneously in water)

RN 152848-09-4 HCAPLUS

Cyclohexanediacetic acid (9CI) (CA INDEX NAME)



CN

L3 ANSWER 8 OF 8 HCAPLUS COPYRIGHT 2007 ACS on STN

IT 152848-09-4, Cyclohexanediacetic acid

RL: BIOL (Biological study)
 (nail polishes containing)

RN 152848-09-4 HCAPLUS

CN Cyclohexanediacetic acid (9CI) (CA INDEX NAME)



```
=> e cyclohexanediacetic acid anhydride
E1
                   CYCLOHEXANEDIACETATO/BI
             1
E2
           249
                   CYCLOHEXANEDIACETIC/BI
E3
             0 --> CYCLOHEXANEDIACETIC ACID ANHYDRIDE/BI
                   CYCLOHEXANEDIACETICDIACETATO/BI
E4
             1
                   CYCLOHEXANEDIACETIMIDE/BI
E5
            39
E6
             2
                   CYCLOHEXANEDIACETIMIDES/BI
             2
                   CYCLOHEXANEDIACETIMIDO/BI
E7
                   CYCLOHEXANEDIACETOMETHYLIMIDE/BI
E8
             1
                   CYCLOHEXANEDIACETONITRILE/BI
E9
            10
E10
             1
                   CYCLOHEXANEDIACETOYL/BI
E11
             5
                   CYCLOHEXANEDIACETYL/BI
E12
                   CYCLOHEXANEDIACID/BI
             4
=> s anhydride
        219700 ANHYDRIDE
         33599 ANHYDRIDES
L4
        230564 ANHYDRIDE
                  (ANHYDRIDE OR ANHYDRIDES)
```

=> s 14 (3w) 13

```
L5
             0 L4 (3W) L3
=> sea abb=on plu=on anhydride
        219700 ANHYDRIDE
         33599 ANHYDRIDES
L6
        230564 ANHYDRIDE
                 (ANHYDRIDE OR ANHYDRIDES)
=> s abb=on plu=on monoamide
          1258 MONOAMIDE
           583 MONOAMIDES
          1633 MONOAMIDE
L7
                 (MONOAMIDE OR MONOAMIDES)
=> s abb=on plu=on precipitation+PFT, old, new/ct
         19338 PRECIPITATION+PFT, OLD, NEW/CT (2 TERMS)
=> s abb=on plu=on acidification+pft, new, old/ct
             2 ACIDIFICATION+PFT, NEW, OLD/CT (1 TERM)
L9
=> s abb=on plu=on ammonia
        214357 AMMONIA
           156 AMMONIAS
L10
        214418 AMMONIA
                  (AMMONIA OR AMMONIAS)
=> s "hydrochloric acid"+pft, old, new/ct
             O "HYDROCHLORIC ACID"+PFT, OLD, NEW/CT (2 TERMS)
L11
=> e hydrochloric acid
                   HYDROCHLORIATE/BI
E1
             2
                  HYDROCHLORIC/BI
E2
        104429
E3
             0 --> HYDROCHLORIC ACID/BI
E4
             2
                  HYDROCHLORICA/BI
E5
            12
                   HYDROCHLORICACID/BI
            10
                   HYDROCHLORICE/BI
E6
                   HYDROCHLORICF/BI
E7
             1
                   HYDROCHLORICI/BI
E8
             1
            1
E9
                   HYDROCHLORICOR/BI
            1
                   HYDROCHLORICS/BI
E10
            18
E11
                  HYDROCHLORICUM/BI
E12
            1
                  HYDROCHLORICUS/BI
=> s e5
L12
            12 HYDROCHLORICACID/BI
=> s abb=on plu=on "acetic anhdride"+pft, new, old/ct
"ACETIC ANHDRIDE" NOT IN RELATIONSHIP FILE
RELATIONSHIP CODE ', NEW, OLD' IGNORED
             0 "ACETIC ANHDRIDE"+PFT, NEW, OLD/CT (1 TERM)
L13
=> e acetic anhydride
                   ACETIATE/BI
El
             3
        247045
                   ACETIC/BI
E2
             0 --> ACETIC ANHYDRIDE/BI
E3
E4
             2
                   ACETIC2/BI
E5
             8
                   ACETICA/BI
E6
             1
                   ACETICACETATE/BI
E7
             3
                   ACETICACI/BI
```

```
E8
            34
                   ACETICACID/BI
E9
                   ACETICACIDETHYL/BI
             2
E10
                  ACETICACIDMONOHYDROCHLORIDE/BI
             1
E11
                  ACETICACIDSHOWED/BI
             1
E12
             1
                  ACETICACIDTERT/BI
=> s acetic
        247045 ACETIC
            22 ACETICS
        247054 ACETIC
L14
                 (ACETIC OR ACETICS)
=> d his
     (FILE 'HOME' ENTERED AT 11:48:54 ON 29 AUG 2007)
     FILE 'STNGUIDE' ENTERED AT 11:49:06 ON 29 AUG 2007
     FILE 'HCAPLUS' ENTERED AT 11:50:33 ON 29 AUG 2007
Ll
              O SEA ABB=ON PLU=ON "CYCLOHEXANEDIACETIC ACID"+PFT, OLD, NEW/CT
     FILE 'REGISTRY' ENTERED AT 11:55:02 ON 29 AUG 2007
                E CYCLOHEXANEDIACETIC/CN
     FILE 'HCAPLUS' ENTERED AT 11:55:03 ON 29 AUG 2007
     FILE 'REGISTRY' ENTERED AT 11:56:31 ON 29 AUG 2007
L2
              1 S E4/CN
     FILE 'HCAPLUS' ENTERED AT 11:56:31 ON 29 AUG 2007
L3
              8 S L2
                E CYCLOHEXANEDIACETIC ACID ANHYDRIDE
L4
         230564 S ANHYDRIDE
L5
              0 S L4 (3W) L3
         230564 SEA ABB=ON PLU=ON ANHYDRIDE
L6
L7
           1633 S ABB=ON PLU=ON MONOAMIDE
L8
          19338 S ABB=ON PLU=ON PRECIPITATION+PFT, OLD, NEW/CT
L9
              2 S ABB=ON PLU=ON ACIDIFICATION+PFT, NEW, OLD/CT
L10
         214418 S ABB=ON PLU=ON AMMONIA
              O S "HYDROCHLORIC ACID"+PFT, OLD, NEW/CT
L11
                E HYDROCHLORIC ACID
L12
             12 S E5
L13
              O S ABB=ON PLU=ON "ACETIC ANHDRIDE"+PFT, NEW, OLD/CT
                E ACETIC ANHYDRIDE
         247054 S ACETIC
L14
=> s 114 (1w) 14
         26509 L14 (1W) L4
L15
=> s abb=on plu=on synth or shythe? or preparation or formation or product? or form
or forming orforms or formed or prep
           326 SYNTH
             2 SHYTHE?
       1538677 PREPARATION
         80195 PREPARATIONS
       1614792 PREPARATION
                  (PREPARATION OR PREPARATIONS)
       2837881 PREPN
```

```
210301 PREPNS
       2996287 PREPN
                 (PREPN OR PREPNS)
       3838782 PREPARATION
                 (PREPARATION OR PREPN)
       2790557 FORMATION
         55019 FORMATIONS
       2821463 FORMATION
                 (FORMATION OR FORMATIONS)
       2950325 PRODUCT?
       1642545 FORM
        621152 FORMS
       2114356 FORM
                 (FORM OR FORMS)
        660407 FORMING
            38 FORMINGS
        660423 FORMING
                 (FORMING OR FORMINGS)
             0 ORFORMS
             O FORMING ORFORMS
                 (FORMING (W) ORFORMS)
       1541162 FORMED
             3 FORMEDS
       1541164 FORMED
                 (FORMED OR FORMEDS)
        132209 PREP
          2288 PREPS
        134283 PREP
                 (PREP OR PREPS)
SYSTEM LIMITS EXCEEDED - SEARCH ENDED
The search profile you entered was too complex or gave too many
answers. Simplify or subdivide the query and try again. If you have
exceeded the answer limit, enter DELETE HISTORY at an arrow prompt
(=>) to remove all previous answers sets and begin at L1. Use the
SAVE command to store any important profiles or answer sets before
using DELETE HISTORY.
=> s abb=ob plu=on synth or synthe? or preparation or produc? or prep
'ABB=OB' IS NOT VALID HERE
The SET parameter ABBREVIATION can be set to values 'ON' or 'OFF'.
For more information enter, "HELP SET ABBREVIATION" at an arrow
prompt (=>).
=> s abb=on plu=on synth or synthe? or prepartion or produc? or prep
           326 SYNTH
       2222349 SYNTHE?
            19 PREPARTION
             3 PREPARTIONS
            22 PREPARTION
                 (PREPARTION OR PREPARTIONS)
       4566040 PRODUC?
       1026837 PRODN
           533 PRODNS
       1027020 PRODN
                 (PRODN OR PRODNS)
       5060028 PRODUC?
                 (PRODUC? OR PRODN)
        132209 PREP
```

2288 PREPS

```
134283 PREP
```

(PREP OR PREPS)

L16 6805049 SYNTH OR SYNTHE? OR PREPARTION OR PRODUC? OR PREP 95% OF LIMIT FOR TOTAL ANSWERS REACHED

=> d his

(FILE 'HOME' ENTERED AT 11:48:54 ON 29 AUG 2007)

FILE 'STNGUIDE' ENTERED AT 11:49:06 ON 29 AUG 2007

FILE 'HCAPLUS' ENTERED AT 11:50:33 ON 29 AUG 2007
L1 0 SEA ABB=ON PLU=ON "CYCLOHEXANEDIACETIC ACID"+PFT, OLD, NEW/CT

FILE 'REGISTRY' ENTERED AT 11:55:02 ON 29 AUG 2007 E CYCLOHEXANEDIACETIC/CN

FILE 'HCAPLUS' ENTERED AT 11:55:03 ON 29 AUG 2007 S E4

FILE 'REGISTRY' ENTERED AT 11:56:31 ON 29 AUG 2007 L2 1 S E4/CN

FILE 'HCAPLUS' ENTERED AT 11:56:31 ON 29 AUG 2007

L3 8 S L2

L7

L8

E CYCLOHEXANEDIACETIC ACID ANHYDRIDE

L4 230564 S ANHYDRIDE

L5 0 S L4 (3W) L3

L6 230564 SEA ABB=ON PLU=ON ANHYDRIDE

1633 S ABB=ON PLU=ON MONOAMIDE

19338 S ABB=ON PLU=ON PRECIPITATION+PFT, OLD, NEW/CT

L9 2 S ABB=ON PLU=ON ACIDIFICATION+PFT, NEW, OLD/CT

L10 214418 S ABB=ON PLU=ON AMMONIA

L11 0 S "HYDROCHLORIC ACID"+PFT, OLD, NEW/CT

E HYDROCHLORIC ACID

L12 12 S E5

L13 0 S ABB=ON PLU=ON "ACETIC ANHDRIDE"+PFT, NEW, OLD/CT

E ACETIC ANHYDRIDE

L14 247054 S ACETIC

L15 26509 S L14 (1W) L4

L16 6805049 S ABB=ON PLU=ON SYNTH OR SYNTHE? OR PREPARTION OR PRODUC? OR PR

=> s abb=on plu=on amination=pft, old, new/ct
NUMERIC VALUE NOT VALID 'PFT, OLD, NEW'
Numeric values may contain 1-8 significant figures. If range notation is used, both the beginning and the end of the range must be specified, e.g., '250-300/MW'. Expressions such as '250-/MW' are not allowed. To search for values above or below a given number, use the >, =>, <, or <= operators, e.g., 'MW => 250'. Text terms cannot be used in numeric expressions. If you specify a unit, it must be dimensionally correct for that field code. To see the unit designations for field codes in the current file, enter "DISPLAY UNIT ALL" at an arrow prompt (=>).

=> s abb=on plu=on amination+pft, old, new, rt/ct L17 157279 AMINATION+PFT, OLD, NEW, RT/CT (6 TERMS)

=> e us4024175/pn

E1 1 US4024173/PN

US4024174/PN Ę2 E3 1 --> US4024175/PN US4024176/PN US4024177/PN US4024178/PN E4 1 E5 1 E6 1 1 US4024179/PN E7 1 US4024181/PN E8 1 US4024182/PN E9 2 US4024183/PN E10 US4024185/PN E11 1 US4024186/PN E12

=> s e3

L18 1 US4024175/PN

- => select rn l1 1
- L1 HAS NO ANSWERS

An L-number has no answers for one of five reasons:

- 1. It is a query that has not been searched, or
- 2. It is the result of a search with zero answers, or
- 3. It is an intermediate result of the ACTIVATE command, or
- 4. It is an intermediate result in SEARCH STEPS, or
- 5. It is an L-number created by the RUN command
- => select rn l18 1
- E1 THROUGH E11 ASSIGNED

=> file reg

COST IN U.S. DOLLARS SINCE FILE ENTRY SE

FULL ESTIMATED COST

SINCE FILE TOTAL ENTRY SESSION 149.60 179.18

FILE 'REGISTRY' ENTERED AT 12:26:48 ON 29 AUG 2007 USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT. PLEASE SEE "HELP USAGETERMS" FOR DETAILS. COPYRIGHT (C) 2007 American Chemical Society (ACS)

Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.

STRUCTURE FILE UPDATES: 28 AUG 2007 HIGHEST RN 945714-55-6 DICTIONARY FILE UPDATES: 28 AUG 2007 HIGHEST RN 945714-55-6

New CAS Information Use Policies, enter HELP USAGETERMS for details.

TSCA INFORMATION NOW CURRENT THROUGH June 29, 2007

Please note that search-term pricing does apply when conducting SmartSELECT searches.

REGISTRY includes numerically searchable data for experimental and predicted properties as well as tags indicating availability of experimental property data in the original document. For information on property searching in REGISTRY, refer to:

http://www.cas.org/support/stngen/stndoc/properties.html

=> s e1-e11

1 1010-26-0/BI

(1010-26-0/RN) 1 4432-19-3/BI

(4432-19-3/RN)

1 5662-95-3/BI (5662-95-3/RN)

(3002-33-37 KM)

1 60142-94-1/BI

(60142-94-1/RN)

1 60142-95-2/BI

(60142-95-2/RN)

1 60142-96-3/BI

(60142-96-3/RN)

1 60142-97-4/BI

(60142-97-4/RN)

1 60142-98-5/BI

(60142-98-5/RN)

1 60142-99-6/BI

(60142-99-6/RN)

1 60143-00-2/BI

(60143-00-2/RN)

1 60175-04-4/BI

(60175-04-4/RN)

L19 11 (1010-26-0/BI OR 4432-19-3/BI OR 5662-95-3/BI OR 60142-94-1/BI OR 60142-95-2/BI OR 60142-96-3/BI OR 60142-97-4/BI OR 60142-98-5 /BI OR 60142-99-6/BI OR 60143-00-2/BI OR 60175-04-4/BI)

=> d scn

L19 ANSWER 1 OF 11 REGISTRY COPYRIGHT 2007 ACS on STN

CN Cyclohexaneacetic acid, 1-(aminomethyl)-, ethyl ester, hydrochloride (9CI) (CA INDEX NAME)

## => d scan

L19 11 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN

IN 1,1-Cyclohexanediacetic acid, monomethyl ester (9CI)

MF C11 H18 O4

CI COM

\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

HOW MANY MORE ANSWERS DO YOU WISH TO SCAN? (1):11

L19 11 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN IN 3-Oxaspiro[5.6]dodecane-2,4-dione (9CI)

MF C11 H16 O3

\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

L19 11 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Cyclohexaneacetic acid, 1-(aminomethyl)-, ethyl ester, hydrochloride (9CI)
MF C11 H21 N O2 . Cl H

HCl

L19 11 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN IN Cyclopentaneacetic acid, 1-(aminomethyl)- (9CI) MF C8 H15 N O2 CI COM

\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

L19 11 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN
IN Cycloheptaneacetic acid, 1-(aminomethyl)-, hydrochloride (9CI)
MF C10 H19 N O2 . Cl H

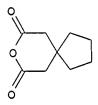
● HCl

L19 11 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN IN Cyclohexaneacetic acid, 1-(aminomethyl)-, hydrochloride (1:1) MF C9 H17 N O2 . Cl H

● HCl

\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

L19 11 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN IN 8-Oxaspiro[4.5]decane-7,9-dione MF C9 H12 O3 CI COM



\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

L19 11 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN IN 3-Oxaspiro[5.5]undecane-2,4-dione (9CI) MF C10 H14 O3

# \*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

L19 11 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN IN Cyclopentaneacetic acid, 1-(2-amino-2-oxoethyl)- (9CI) MF C9 H15 N O3

# \*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

L19 11 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN IN 1,1-Cycloheptanediacetic acid, monomethyl ester (9CI) MF C12 H20 O4

# \*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

L19 11 ANSWERS REGISTRY COPYRIGHT 2007 ACS on STN IN Cyclohexaneacetic acid, 1-(aminomethyl)-

MF C9 H17 N O2

CI COM

```
СH<sub>2</sub>- NH<sub>2</sub>
СH<sub>2</sub>- CO<sub>2</sub>H
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## \*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

# ALL ANSWERS HAVE BEEN SCANNED

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=> d his
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L2

L7

(FILE 'HOME' ENTERED AT 11:48:54 ON 29 AUG 2007)

FILE 'STNGUIDE' ENTERED AT 11:49:06 ON 29 AUG 2007

FILE 'HCAPLUS' ENTERED AT 11:50:33 ON 29 AUG 2007

L1 0 SEA ABB=ON PLU=ON "CYCLOHEXANEDIACETIC ACID"+PFT, OLD, NEW/CT

FILE 'REGISTRY' ENTERED AT 11:55:02 ON 29 AUG 2007 E CYCLOHEXANEDIACETIC/CN

FILE 'HCAPLUS' ENTERED AT 11:55:03 ON 29 AUG 2007 S E4

FILE 'REGISTRY' ENTERED AT 11:56:31 ON 29 AUG 2007 1 S E4/CN

FILE 'HCAPLUS' ENTERED AT 11:56:31 ON 29 AUG 2007

L3 8 S L2

E CYCLOHEXANEDIACETIC ACID ANHYDRIDE

L4 230564 S ANHYDRIDE

L5 0 S L4 (3W) L3

L6 230564 SEA ABB=ON PLU=ON ANHYDRIDE

1633 S ABB=ON PLU=ON MONOAMIDE

L8 19338 S ABB=ON PLU=ON PRECIPITATION+PFT, OLD, NEW/CT

L9 2 S ABB=ON PLU=ON ACIDIFICATION+PFT, NEW, OLD/CT

L10 214418 S ABB=ON PLU=ON AMMONIA

L11 0 S "HYDROCHLORIC ACID"+PFT, OLD, NEW/CT

E HYDROCHLORIC ACID

L12 12 S E5

L13 0 S ABB=ON PLU=ON "ACETIC ANHDRIDE"+PFT, NEW, OLD/CT

E ACETIC ANHYDRIDE

L14 247054 S ACETIC

L15 26509 S L14 (1W) L4

L16 6805049 S ABB=ON PLU=ON SYNTH OR SYNTHE? OR PREPARTION OR PRODUC? OR PR

L17 157279 S ABB=ON PLU=ON AMINATION+PFT, OLD, NEW, RT/CT

E US4024175/PN

L18 1 S E3

SELECT RN L18 1

FILE 'REGISTRY' ENTERED AT 12:26:48 ON 29 AUG 2007 L19 11 S E1-E11

=> s (1,1-cyclohexanediacetic acid)+rtcs, new, old/ct MISSING OPERATOR

=> s abb=on plu=on "1,1-cyclohexanediacetic acid"+rtcs, new, old/ct 'CT' IS NOT A VALID FIELD CODE

L20 0 "1,1-CYCLOHEXANEDIACETIC ACID"+RTCS, NEW, OLD/CT

=> s abb=on plu=on gabapentine+rtcs, new, old, pft/ct

'CT' IS NOT A VALID FIELD CODE

L21 0 GABAPENTINE+RTCS, NEW, OLD, PFT/CT

=> s gabapentin

L22 7 GABAPENTIN

=> s 122 and 12

L23 0 L22 AND L2

=> s 122 and 13

L24 0 L22 AND L3

=> file hcaplus

COST IN U.S. DOLLARS

SINCE FILE TOTAL

ENTRY SESSION 17.25 196.43

FULL ESTIMATED COST

FILE 'HCAPLUS' ENTERED AT 12:40:47 ON 29 AUG 2007 USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT. PLEASE SEE "HELP USAGETERMS" FOR DETAILS. COPYRIGHT (C) 2007 AMERICAN CHEMICAL SOCIETY (ACS)

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FILE COVERS 1907 - 29 Aug 2007 VOL 147 ISS 10 FILE LAST UPDATED: 28 Aug 2007 (20070828/ED)

New CAS Information Use Policies, enter HELP USAGETERMS for details.

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s gabapentin

1956 GABAPENTIN

1 GABAPENTINS

L25 1956 GABAPENTIN

(GABAPENTIN OR GABAPENTINS)

=> s 125 and 13

L26 0 L25 AND L3

=> d his

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(FILE 'HOME' ENTERED AT 11:48:54 ON 29 AUG 2007)
     FILE 'STNGUIDE' ENTERED AT 11:49:06 ON 29 AUG 2007
     FILE 'HCAPLUS' ENTERED AT 11:50:33 ON 29 AUG 2007
L1
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     FILE 'REGISTRY' ENTERED AT 11:55:02 ON 29 AUG 2007
               E CYCLOHEXANEDIACETIC/CN
     FILE 'HCAPLUS' ENTERED AT 11:55:03 ON 29 AUG 2007
                S E4
     FILE 'REGISTRY' ENTERED AT 11:56:31 ON 29 AUG 2007
L2
              1 S E4/CN
     FILE 'HCAPLUS' ENTERED AT 11:56:31 ON 29 AUG 2007
L3
              8 S L2
               E CYCLOHEXANEDIACETIC ACID ANHYDRIDE
         230564 S ANHYDRIDE
L4
L5
              0 S L4 (3W) L3
L6
         230564 SEA ABB=ON PLU=ON ANHYDRIDE
L7
          1633 S ABB=ON PLU=ON MONOAMIDE
         19338 S ABB=ON PLU=ON PRECIPITATION+PFT, OLD, NEW/CT
              2 S ABB=ON PLU=ON ACIDIFICATION+PFT, NEW, OLD/CT
L9
L10
         214418 S ABB=ON PLU=ON AMMONIA
              O S "HYDROCHLORIC ACID"+PFT, OLD, NEW/CT
L11
                E HYDROCHLORIC ACID
             12 S E5
L12
              O S ABB=ON PLU=ON "ACETIC ANHDRIDE"+PFT, NEW, OLD/CT
L13
                E ACETIC ANHYDRIDE
L14
         247054 S ACETIC
L15
          26509 S L14 (1W) L4
        6805049 S ABB=ON PLU=ON SYNTH OR SYNTHE? OR PREPARTION OR PRODUC? OR PR
L16
        157279 S ABB=ON PLU=ON AMINATION+PFT, OLD, NEW, RT/CT
L17
               E US4024175/PN
              1 S E3
L18
                SELECT RN L18 1
     FILE 'REGISTRY' ENTERED AT 12:26:48 ON 29 AUG 2007
L19
             11 S E1-E11
              O S ABB=ON PLU=ON "1,1-CYCLOHEXANEDIACETIC ACID"+RTCS, NEW, OLD/C
L20
L21
             O S ABB=ON PLU=ON GABAPENTINE+RTCS, NEW, OLD, PFT/CT
L22
             7 S GABAPENTIN
L23
              0 S L22 AND L2
L24
              0 S L22 AND L3
     FILE 'HCAPLUS' ENTERED AT 12:40:47 ON 29 AUG 2007
           1956 S GABAPENTIN
L25
             0 S L25 AND L3
L26
=> s 125 and 16
           20 L25 AND L6
=> s 127 and 17
             4 L27 AND L7
L28
=> d 128 ibib abs
```

L28 ANSWER 1 OF 4 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2005:1026923 HCAPLUS

DOCUMENT NUMBER: 143:286690

TITLE: Synthesis of 4-tert-butylgabapentin

INVENTOR(S): Kuppuswamy, Nagarajan; Hariharan, Sivaramakrishnan; Iyer, Venkatachalam Shankar; Balakrishnan, Suresh Babu; Krishnamurthi, Gopalakrishnan; Kuppana, Ananda;

Karuppiah, Muruga Poopati Raja; Padmanabhan, Balaram; Subrayashastry, Aravinda; Prema, Gouriamma Vasudev;

Narayanaswamy, Shamala

PATENT ASSIGNEE(S): Hikal Limited, India; Indian Institute of Science

SOURCE: PCT Int. Appl., 17 pp. CODEN: PIXXD2

DOCUMENT TYPE: Patent LANGUAGE: English

FAMILY ACC. NUM. COUNT: 3

PATENT INFORMATION:

	PATENT NO.							KIND DATE						DATE					
							-												
	WO 2	005	0877	09		A1	;	2005	0922	1	WO 20	005-	IN82		20050316				
		W :	ΑE,	AG,	AL,	AM,	ΑT,	AU,	AZ,	BA,	BB,	BG,	BR,	BW,	BY,	BZ,	CA,	CH,	
			CN,	CO,	CR,	CU,	CZ,	DE,	DK,	DM,	DZ,	EC,	EE,	EG,	ES,	FI,	GB,	GD,	
			GE,	GH,	GM,	HR,	HU,	ID,	IL,	IN,	IS,	JP,	KE,	KG,	KP,	KR,	ΚŻ,	LC,	
			LK,	LR,	LS,	LT,	LU,	LV,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	MZ,	NA,	NI,	
			NO,	NZ,	OM,	PG,	PH,	PL,	PT,	RO,	RU,	SC,	SD,	SE,	SG,	SK,	SL,	SM,	
			SY,	TJ,	TM,	TN,	TR,	TT,	TZ,	UA,	ΰĠ,	US,	UΖ,	VC,	VN,	YU,	ZA,	ZM,	ZW
		RW:	BW,	GH,	GM,	KE,	LS,	MW,	MZ,	NA,	SD,	SL,	SZ,	TZ,	UG,	ZM,	ZW,	AM,	
			ΑZ,	BY,	KG,	KZ,	MD,	RU,	TJ,	TM,	AT,	ΒĒ,	BG,	CH,	CY,	CZ,	DE,	DK,	
			EE,	ES,	FI,	FR,	GB,	GR,	HU,	IE,	IS,	IT,	LT,	LU,	MC,	NL,	PL,	PT,	
			RO,	SE,	SI,	SK,	TR,	BF,	ВJ,	CF,	CG,	CI,	CM,	GA,	GN,	GQ,	GW,	ML,	
			MR,	ΝĒ,	SN,	TD,	TG												
	CA 2	570	255			A1		2005	0922	(	CA 2	005-	2570	255	20050316				
	EP 1	763	503			A1		2007	0321	:	EP 2	005-	7329	85	20050316				
		R:	AT,	BE,	BG,	CH,	CY,	CZ,	DE,	DK,	EE,	ES,	FI,	FR,	GB,	GR,	HU,	IE,	
								MC,											
PRIO	RITY	APP:								US 2004-553565P									
										1	WO 2	005-	IN82		W 20050316				
										_									-

The invention relates to a process for the preparation of the cis (Z) and trans (E) stereoisomers of 4-tert-butylgabapentin. 4-Tert-butylgabapentin as a mixture of stereoisomers was prepared by treating 4-tert-butylcyclohexanone with Et cyanoacetate and ammonia in methanol, hydrolysis of the dicyano imide product with hot sulfuric acid, conversion to the anhydride , treatment with aqueous ammonia to give the monoamide as a mixture of approx. equal proportions of stereoisomers, reaction with NaOBr to form the lactam, hydrolysis of the lactam with hot concentrated HCl, and neutralization with aqueous NaOH to pH 7. The stereoisomers of

4-tert-butylgabapentin were separated by fractional crystallization from aqueous methanol.

REFERENCE COUNT: 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

## => d 128 2-4 ibib abs

L28 ANSWER 2 OF 4 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2005:1026620 HCAPLUS

DOCUMENT NUMBER: 143:267241

TITLE: Preparation of gabapentin analogues

Krishnamurthi, Gopalakrishnan; Kuppanna, Ananda; Karuppiah, Muruga Poopati Raja; Padmanabhan, Balaram;

Subrayashastry, Aravinda; Prema, Gouriamma Vasudev;

Narayanaswamy, Shamala

PATENT ASSIGNEE(S): Hikal Limited, India; Indian Institute of Science

SOURCE: U.S. Pat. Appl. Publ., 6 pp.

CODEN: USXXCO

DOCUMENT TYPE: Patent LANGUAGE: English

FAMILY ACC. NUM. COUNT: 3

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.		DATE
US 2005209332	A1	20050922	US 2005-79481		20050315
US 2007123591	Al	20070531	US 2006-583953		20061020
PRIORITY APPLN. INFO.:		•	US 2004-553565P	P	20040317
			US 2005-79481	A2	20050315

OTHER SOURCE(S): CASREACT 143:267241

The invention relates to a process for the preparation of the cis (Z) and trans (E) stereoisomers of 4-tert-butylgabapentin. 4-Tert-butylgabapentin as a mixture of stereoisomers was prepared by treating 4-tert-butylcyclohexanone with Et cyanoacetate and ammonia in methanol, hydrolysis of the dicyano imide product with hot sulfuric acid, conversion to the anhydride , treatment with aqueous ammonia to give the monoamide as a mixture of approx. equal proportions of stereoisomers, reaction with NaOBr to form the lactam, hydrolysis of the lactam with hot concentrated HCl, and neutralization with aqueous NaOH to pH 7. The stereoisomers of

4-tert-butylgabapentin were separated by fractional crystallization from aqueous methanol.

L28 ANSWER 3 OF 4 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2005:426561 HCAPLUS

DOCUMENT NUMBER: 142:463372

TITLE: Process for the preparation of gabapentin

via the Hoffmann rearrangement of 1,1-

cyclohexanediacetic acid monoamide

INVENTOR(S): Arrighi, Katiuscia; Cannata, Vincenzo; Corcella,

OR(5): Alight, Rational Cabana Nicelia, and an

Francesco; Marchioro, Gaetano; Nicoli, Andrea;

Paiocchi, Maurizio; Villa, Marco

PATENT ASSIGNEE(S): Zambon Group S.p.A., Italy

SOURCE: PCT Int. Appl., 11 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND DATE	APPLICATION NO.	DATE
WO 2005044779	A2 20050519	WO 2004-EP52894	20041109
WO 2005044779	A3 20050714		
W: AE, AG, AL,	AM, AT, AU, AZ,	BA, BB, BG, BR, BW, BY	, BZ, CA, CH,
CN, CO, CR,	CU, CZ, DE, DK,	DM, DZ, EC, EE, EG, ES	, FI, GB, GD,
		IN, IS, JP, KE, KG, KF	
LK, LR, LS,	LT, LU, LV, MA,	MD, MG, MK, MN, MW, MX	K, MZ, NA, NI,

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NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY,
            TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW
        RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM,
            AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK,
            EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LU, MC, NL, PL, PT, RO,
            SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR,
            NE, SN, TD, TG
                                                                 20041109
     CA 2543275
                         Al
                               20050519
                                         CA 2004-2543275
                         A2
                                          EP 2004-804523
     EP 1682488
                               20060726
                                                                 20041109
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             IS, YU
                                           JP 2006-538854
                               20070426
     JP 2007510695
                                                                  20041109
                               20070608
                                           IN 2006-CN1621
     IN 2006CN01621
                         Α
                                                                  20060510
     US 2007066843
                         A1
                               20070322
                                           US 2006-578783
                                                                  20061206
                                                           A 20031111
W 20041109
PRIORITY APPLN. INFO.:
                                           IT 2003-MI2165
                                           WO 2004-EP52894
OTHER SOURCE(S):
                        CASREACT 142:463372
    Gabapentin and its salts (e.g., gabapentin
AB
     hydrochloride) are prepared by the Hoffmann rearrangement of
     1,1-cyclohexanediacetic acid monoamide, prepared by the
     monoamidation of 1,1-cyclohexanediacetic anhydride with aqueous
     ammonia, optionally followed by salification in the case of required salt
     formation.
L28 ANSWER 4 OF 4 HCAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER:
                        2003:22835 HCAPLUS
DOCUMENT NUMBER:
                        138:73019
                        Amidation process for the preparation of
TITLE:
                        1,1-cyclohexanediacetic acid monoamide from
                        1,1-cyclohexanediacetic anhydride and
                        aqueous ammonia
                        Oren, Jacob
INVENTOR(S):
PATENT ASSIGNEE(S):
                        Bromine Compounds Ltd., Israel
SOURCE:
                        PCT Int. Appl., 15 pp.
                        CODEN: PIXXD2
DOCUMENT TYPE:
                        Patent
                        English
LANGUAGE:
FAMILY ACC. NUM. COUNT:
                        1
PATENT INFORMATION:
                       KIND DATE APPLICATION NO.
                                                                DATE
     PATENT NO.
     WO 2003002517 A1 20030109 WO 2002-IL473
                                                                 20020617
        W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
             CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
             GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR,
             LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH,
             PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ,
             UA, UG, US, UZ, VN, YU, ZA, ZM, ZW
         RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, CH,
             CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR,
             BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG
                               20030303
                                           AU 2002-311607
                                                                 20020617
     AU 2002311607
                        A1
                                                           A 20010628
W 20020617
                                           IL 2001-144066
PRIORITY APPLN. INFO.:
                                           WO 2002-IL473
```

OTHER SOURCE(S): CASREACT 138:73019

AB 1,1-Cyclohexanediacetic acid monoamide (CHDAAM), a gabapentin intermediate (no data), is prepared in high yield and

```
selectivity by amination of 1,1-cyclohexanediacetic anhydride
     (CDAAn) with aqueous ammonia, followed by neutralization of the reaction
mixture
     with an acid (e.g., H2SO4) such that crude CHDAAM is precipitated, filtered,
and
    purified by crystallization from a solvent. The amination is carried out at
     <20° with aqueous ammonia having a concentration of 25-35% and in a molar
     ratio, relative to the CHDAAn, of 5-10, resp. The neutralization is
     carried out with an aqueous solution of H2SO4 having a concentration of 30-70%
and is
     continued until a slightly acid solution is obtained.
                               THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS
REFERENCE COUNT:
                         2
                               RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT
=> s 1,1-cyclohexanediacetic acid
       9264151 1
       9264151 1
           249 CYCLOHEXANEDIACETIC
       4427414 ACID
       1588937 ACIDS
       4929386 ACID
                 (ACID OR ACIDS)
L29
            99 1,1-CYCLOHEXANEDIACETIC ACID
                 (1 (W) 1 (W) CYCLOHEXANEDIACETIC (W) ACID)
=> d his
     (FILE 'HOME' ENTERED AT 11:48:54 ON 29 AUG 2007)
     FILE 'STNGUIDE' ENTERED AT 11:49:06 ON 29 AUG 2007
     FILE 'HCAPLUS' ENTERED AT 11:50:33 ON 29 AUG 2007
              O SEA ABB=ON PLU=ON "CYCLOHEXANEDIACETIC ACID"+PFT, OLD, NEW/CT
Ll
     FILE 'REGISTRY' ENTERED AT 11:55:02 ON 29 AUG 2007
                E CYCLOHEXANEDIACETIC/CN
     FILE 'HCAPLUS' ENTERED AT 11:55:03 ON 29 AUG 2007
                S E4
     FILE 'REGISTRY' ENTERED AT 11:56:31 ON 29 AUG 2007
L2
              1 S E4/CN
     FILE 'HCAPLUS' ENTERED AT 11:56:31 ON 29 AUG 2007
L3
                E CYCLOHEXANEDIACETIC ACID ANHYDRIDE
         230564 S ANHYDRIDE
L4
L5
              0 S L4 (3W) L3
L6
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           1633 S ABB=ON PLU=ON MONOAMIDE
L7
          19338 S ABB=ON PLU=ON PRECIPITATION+PFT, OLD, NEW/CT
L8
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O S ABB=ON PLU=ON "ACETIC ANHDRIDE"+PFT, NEW, OLD/CT

2 S ABB=ON PLU=ON ACIDIFICATION+PFT, NEW, OLD/CT

O S "HYDROCHLORIC ACID"+PFT, OLD, NEW/CT

214418 S ABB=ON PLU=ON AMMONIA

12 S E5

E HYDROCHLORIC ACID

E ACETIC ANHYDRIDE

L9

L10

L11

L12 L13

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247054 S ACETIC
L14
1.15
        26509 S L14 (1W) L4
       6805049 S ABB=ON PLU=ON SYNTH OR SYNTHE? OR PREPARTION OR PRODUC? OR PR
L16
       157279 S ABB=ON PLU=ON AMINATION+PFT, OLD, NEW, RT/CT
L17
               E US4024175/PN
L18 .
             1 S E3
               SELECT RN L18 1
    FILE 'REGISTRY' ENTERED AT 12:26:48 ON 29 AUG 2007
L19
           11 S E1-E11
             0 S ABB=ON PLU=ON "1,1-CYCLOHEXANEDIACETIC ACID"+RTCS, NEW, OLD/C
L20
             O S ABB=ON PLU=ON GABAPENTINE+RTCS, NEW, OLD, PFT/CT
L21
L22
             7 S GABAPENTIN
             0 S L22 AND L2
L23
             0 S L22 AND L3
L24
    FILE 'HCAPLUS' ENTERED AT 12:40:47 ON 29 AUG 2007
      1956 S GABAPENTIN
L25
            0 S L25 AND L3
L26
L27
            20 S L25 AND L6
L28
            4 S L27 AND L7
            99 S 1,1-CYCLOHEXANEDIACETIC ACID
L29
=> s 129 (3w) 16
            4 L29 (3W) L6
=> s 130 not 128
L31
            3 L30 NOT L28
=> d 131 1-3 ibib abs
L31 ANSWER 1 OF 3 HCAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 2001:930766 HCAPLUS
DOCUMENT NUMBER:
                        136:19880
                       Preparation of 1-(2-amino-2-oxoethyl)cyclohexaneacetic
TITLE:
                        acid
INVENTOR(S):
                        Tang, Miaorong; Fan, Weirong; Liu, Tianchun; Zhang,
                        Xiaobo
                       Hangzhou Shouxin Fine Chemical Co., Ltd., Peop. Rep.
PATENT ASSIGNEE(S):
                        China
SOURCE:
                        Faming Zhuanli Shenqing Gongkai Shuomingshu, 5 pp.
                        CODEN: CNXXEV
DOCUMENT TYPE:
                        Patent
                        Chinese
LANGUAGE:
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:
                   KIND DATE APPLICATION NO.
     PATENT NO.
                                                               DATE
                       ----
                                         ______
                        A 20010606
                                         CN 2000-128111
                                                                20001201
     CN 1297885
     CN 1109017
                       В
                             20030521
PRIORITY APPLN. INFO.:
                                          CN 2000-128111
                                                                20001201
                       CASREACT 136:19880
OTHER SOURCE(S):
    1-(2-Amino-2-oxoethyl)cyclohexaneacetic acid is synthesized by condensing
AB
     cyclohexanone with Et cyanoacetate in ethanol under bubbling NH3 for 18-26
     h, stirring at 0° for 18-26 h and at 25° for 100-130 h to
     obtain \alpha, \alpha-dicyano-1,1-cyclohexanediacetimide ammonium salt,
```

hydrolyzing with H2SO4 solution at 200° for 30 min to obtain 1

,1-cyclohexanediacetic acid, dehydrating

with acetic anhydride to obtain 1,1-cyclohexanediacetic anhydride, aminolyzing with NH3 or NH4OH at 30-110° for 3-8 h, and recrystg. with ethanol.

L31 ANSWER 2 OF 3 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1988:186295 HCAPLUS

DOCUMENT NUMBER: 108:186295

TITLE: Studies in the cycloheptane series. Part XXX.

Friedel-Crafts reaction of the anhydride of 3,4-dimethylcyclohexane-1,1-diacetic acid with

aromatic hydrocarbons and synthesis of 2,3-dimethyl-9,10-benzo-substituted

benzospiro[5.6] dodecanes

AUTHOR(S): Gautam, R. K.; Kannan, S.; Saharia, G. S.

Ι

ΙI

CORPORATE SOURCE: Dep. Chem., Univ. Delhi, Delhi, 110007, India

SOURCE: Journal of the Institution of Chemists (India) (1987),

59(2), 95-9

CODEN: JOICA7; ISSN: 0020-3254

DOCUMENT TYPE: Journal LANGUAGE: English

OTHER SOURCE(S): CASREACT 108:186295

GI

$$R^1$$
 $HO_2CCH_2$ 
 $Me$ 
 $R^2$ 
 $C(:Z)CH_2$ 
 $Me$ 

$$R^1$$
 $R^2$ 
 $R^3$ 
 $R^2$ 
 $R^3$ 
 $R^2$ 
 $R^3$ 

Friedel-Crafts reaction of 3,4-dimethyl-1,1cyclohexanediacetic acid anhydride with C6H6,
PhMe, o-, m-, and p-xylene, PhCl, PhOMe, and tetralin gave 70-80% aryl
ketones I [R = R2 = R3 = H, R1 = H, Me, Cl, MeO; RR1 = (CH2)4, R2 = R3 =
H; R = R3 = Me, R1 = R2 = H; R = R3 = H, R1 = R2 = Me; R = R2 = H, R1 = R3
= Me; Z = O]. Clemmensen reduction of I (Z = O) gave I (Z = H2) which were
cyclized with polyphosphoric acid to give 60-70% benzospiro[5.6] dodecanes
II (same R-R3; Z1 = O). Clemmensen reduction of II (Z1 = O) gave II (Z1 =
H2).

L31 ANSWER 3 OF 3 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 1963:66337 HCAPLUS

DOCUMENT NUMBER: 58:66337

ORIGINAL REFERENCE NO.: 58:11294d-h,11295a-e

```
TITLE:
                         Catalytic dehydrogenation. VIII. Synthesis and
                         dehydrogenation of spiro[6.5]dodecanes
AUTHOR(S):
                         Sen Gupta, S. C.; Sen, Parimal Krishna
                         Ramakrishna Mission Vidyamandir, Belur Math, India
CORPORATE SOURCE:
SOURCE:
                         J. Indian Chem. Soc. (1962), 39, 815-22
DOCUMENT TYPE:
                         Journal
LANGUAGE:
                         Unavailable
     For diagram(s), see printed CA Issue.
     cf. ibid. 660; CA 50, 3364h. The synthesis of Ia (R2 = R3 = H) (I, R = R1
AB
     = H) and its alkyl derivs. were described. Ia when heated with Pd-C at
     370-400° in a sealed tube underwent dehydrogenation accompanied by
     ring transformation, providing an anthracene or a phenanthrene as the main
     product. By the method of Ali, et al. (CA 31, 62187), were prepared IIa
     [(R2R3 = )0] (II, R = R1 = H) and IIa (R2 = R3 = H) (III, R = R1 = H), b1
     188°, m. 57-8° (hexane). III (R = R1 = H) (10 g.) and
     polyphosphoric acid (PPA) (from 60 g. P2O5 and 60 ml. 89% H3PO4) heated
     and stirred 1.5 hrs. on a steam bath, poured on crushed ice, and the
     product isolated with Et20 gave 6.5 g. Ia [(R2R3 = )0] (IV R = R1 = H),
     bl 166-8°, m. 58° (hexane); 2,4-dinitrophenylhydrazone m.
     226° (EtOAc). IV (R = R1 = H) (9 g.) gently boiled 24 hrs. with 40
     g. amalgamated Zn and 40 ml. concentrated HCl and the product isolated with
Et20
     gave 6 g. I (R = R1 = H) (IVa), b1 152-3°, d32 0.9986, n32D 1.5445.
     IVa (2.51 g.) heated 18 hrs. at 380-400° with 0.28 g. 10% Pd-C in a
     sealed tube, the product isolated with Et20, and chromatographed on Al203
     with hexane gave initially o-xylene, b. 140-5°, oxidized by
     alkaline KMnO4 to o-C6H4(CO2H)2 (IVb), m. 200° (decomposition)
     (anhydride m. 130°). Later fractions gave anthracene (V) isolated
     via the trinitrobenzene (VI) complex. From 14 g. 1,1-
     cyclohexanediacetic acid anhydride (VII), 70
     ml. PhMe, and 27 g. AlCl3 was prepared as above 21 g. II (R = Me, R1 = H)
     (VIIa), m. 87-8° (EtOH, then hexane); semicarbazone m. 200°
     (decomposition) (EtOH). VIIa heated with alkaline KMnO4 solution gave
p-C6H4 (CO2H) 2
     (VIII); di-Me ester (IX) m. 140°. VIIa (25 g.) heated 24 hrs. with
     100 g. amalgamated Zn and 100 ml. concentrated HCl gave 12 g. III (R = Me, R1 =
     H) (VIIIa), b1 192-4°. VIIIa (8 g.) cyclized with PPA (from 60 g.
     P205 and 40 ml. 89% H3PO4) as above gave IV (R = Me, R1 = H) (VIIIb), b1
     178°, m. 60-1°; 2,4-dinitrophenylhydrazone m. 216-17°
     (EtOAc). VIIIb (6 g.) heated 24 hrs. with 30 g. amalgamated Zn and 30 ml.
     concentrated HCl gave 4 g. I (R = Me, R = H) (VIIIc), b1 173-5°, d32 1.0,
     n32D 1.543. VIIIc (1.77 g.) and 0.2 g. 10% Pd-C heated 16 hrs. at
     380-400° in a sealed tube, the product chromatographed on Al203
     with hexane as above, and the combined oils from the 1st and 2nd eluates
     distilled gave 1,2,4-C6H3Me3, oxidized by alkaline KMnO4 solution to
     1,2,4-C6H3(CO2H)3, m. 216° (decomposition); the 3rd and 4th eluates
     concentrated, each residual solid (small amts.) treated with VI, and the
     combined complexes (m. 124-30°) crystallized repeatedly from EtOH gave
     VI complex of 2-methylanthracene (X), m. 130°, from which was
     regenerated X, m. 201° (EtOH). VII (15 g.) in 20 ml. PhEt added to
     25 g. anhydrous AlCl3 suspended in 75 ml. ice cold dry (Cl2CH)2 and worked up
     as above gave 10 g. II (R = Et, R1 = H), b0.8 210-12°
     [semicarbazone, m. 182-3° (decomposition) (EtOH)], oxidized with alkaline
     KMnO4 solution to VIII, and heated (55 g.) 30 hrs. with 200 g. amalgamated Zn
     and 200 ml. concentrated HCl to 38 g. III (R = Et, R1 = H) (Xa), bl,
     210°. Xa (8.1 g.) cyclized with PPA (from 35 g. P205 and 15 ml.
     89% H3PO4 as above gave 4.19 g. IV (R = Et, R1 = H), b1 185-7^{\circ}
     [semicarbazone, m. 222° (decomposition) (EtOH)], which (10 g.) heated 30
     hrs. with 40 g. amalgamated Zn and 40 ml. concentrated HCl gave 7 g. I (R = Et,
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Serial No.: 10578783 R1 = H) (Xb), b1 165-7°, d32 0.9947, n32D 1.541. Xb (2.45 g.) and 0.25 g. 10% Pd-C heated 16 hrs. at 380-400° in a sealed tube and the product chromatographed on Al203 with hexane as above gave (from the 1st and 2nd eluates) traces unchanged Xb; the 3rd and 4th eluates concentrated, each residual oil treated with VI, and the combined complexes (m. 110-18°) crystallized repeatedly from EtOH gave V complex of 2-ethylanthracene (XI), m. 119-20°, from which was regenerated XI, m. 150-1°. From 48 g. 4-methyl-1,1cyclohexanediacetic acid anhydride, 150 ml. dry C6H5, and 70 g. AlCl3 was prepared 12 g. II (R = H, R1 = Me) (XIa), m. 113° (EtOH, then hexane); from the EtOH mother liquor was isolated 20 g. stereoisomer (XII) of II (R = H, R1 = Me), viscous mass, bl 200-5°. XII (17 g.) heated 36 hrs. with 75 g. amalgamated Zn and 75 ml. concentrated HCl gave 10 g. III (R = H, R1 = Me), bl  $183-5^{\circ}$ , cyclized with PPA (from 30 g. P2O5 and 15 ml. 89% H3PO4) to 6.5 g. IV (R = H, R1 = Me) (XIIa), b1 162-3°; 2,4-dinitrophenylhydrazone, m. 218-19° (EtOAc). XIa reduced with amalgamated Zn and concentrated HCl and the resulting product cyclized with PPA gave XIIa. XIIa (10 g.) gently boiled 24 hrs. with 40 g. amalgamated Zn and 40 ml. concentrated HCl gave 5.9 q. I (R = H, R1 = Me), b1 150-1°, d30 1.0128, n30D 1.5410, which (2.7 g.) and 0.29 g. 10% Pd-C heated 16 hrs. at 380-400° in a sealed tube and the product chromatographed on Al203 with hexane gave (from the 1st, 2nd, and 3rd eluates) o-xylene, b. .apprx.145°, oxidized by alkaline KMnO4 solution to IVb; the 4th, 5th, and 6th eluates concentrated, each residual oil (containing very little solid) treated with VI, and the combined complexes (m. 148-55°) crystallized repeatedly from EtOH gave VI complex of 3-methylphenanthrene (XIII), m. 155°, from which was regenerated XIII, m. 62-3° (EtOH) [picrate, m. 140-1° (EtOH)]. => d his (FILE 'HOME' ENTERED AT 11:48:54 ON 29 AUG 2007) FILE 'STNGUIDE' ENTERED AT 11:49:06 ON 29 AUG 2007

L5

L6

L7

FILE 'HCAPLUS' ENTERED AT 11:50:33 ON 29 AUG 2007 O SEA ABB=ON PLU=ON "CYCLOHEXANEDIACETIC ACID"+PFT, OLD, NEW/CT L1

FILE 'REGISTRY' ENTERED AT 11:55:02 ON 29 AUG 2007 E CYCLOHEXANEDIACETIC/CN

FILE 'HCAPLUS' ENTERED AT 11:55:03 ON 29 AUG 2007 S E4

FILE 'REGISTRY' ENTERED AT 11:56:31 ON 29 AUG 2007 L2 1 S E4/CN

FILE 'HCAPLUS' ENTERED AT 11:56:31 ON 29 AUG 2007

8 S L2 L3

E CYCLOHEXANEDIACETIC ACID ANHYDRIDE

230564 S ANHYDRIDE L4

0 S L4 (3W) L3

230564 SEA ABB=ON PLU=ON ANHYDRIDE

1633 S ABB=ON PLU=ON MONOAMIDE

19338 S ABB=ON PLU=ON PRECIPITATION+PFT, OLD, NEW/CT L8

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L9
              2 S ABB=ON PLU=ON ACIDIFICATION+PFT, NEW, OLD/CT
L10
         214418 S ABB=ON PLU=ON AMMONIA
              O S "HYDROCHLORIC ACID"+PFT, OLD, NEW/CT
L11
                E HYDROCHLORIC ACID
L12
              O S ABB=ON PLU=ON "ACETIC ANHDRIDE"+PFT, NEW, OLD/CT
L13
                E ACETIC ANHYDRIDE
         247054 S ACETIC
L14
L15
          26509 S L14 (1W) L4
        6805049 S ABB=ON PLU=ON SYNTH OR SYNTHE? OR PREPARTION OR PRODUC? OR PR
L16
         157279 S ABB=ON PLU=ON AMINATION+PFT, OLD, NEW, RT/CT
L17
                E US4024175/PN
L18
              1 S E3
                SELECT RN L18 1
     FILE 'REGISTRY' ENTERED AT 12:26:48 ON 29 AUG 2007
L19
             11 S E1-E11
              O S ABB=ON PLU=ON "1,1-CYCLOHEXANEDIACETIC ACID"+RTCS, NEW, OLD/C
L20
              O S ABB=ON PLU=ON GABAPENTINE+RTCS, NEW, OLD, PFT/CT
L21
L22
              7 S GABAPENTIN
              0 S L22 AND L2
L23
L24
              0 S L22 AND L3
     FILE 'HCAPLUS' ENTERED AT 12:40:47 ON 29 AUG 2007
           1956 S GABAPENTIN
L25
             0 S L25 AND L3
L26
             20 S L25 AND L6
L27
             4 S L27 AND L7
L28
             99 S 1,1-CYCLOHEXANEDIACETIC ACID
L29
              4 S L29 (3W) L6
L30
L31
              3 S L30 NOT L28
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L32
             2 L29 AND L17 NOT L28
=> d 132 1-2 ibib abs
L32 ANSWER 1 OF 2 HCAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 2003:678769 HCAPLUS
DOCUMENT NUMBER:
                          139:197197
                          Preparation of new mineral acid addition salts of
TITLE:
                          qabapentin
                          Vittal, Tangirala Venkata Subramanya Krishna; Taj,
INVENTOR(S):
                          Shabbir Ali; Kodimuthali, Armuqam; Maddali, Kasturaiah
                          Shasun Chemicals and Drugs Limited, India
PATENT ASSIGNEE(S):
                          PCT Int. Appl., 18 pp.
SOURCE:
                          CODEN: PIXXD2
DOCUMENT TYPE:
                          Patent
LANGUAGE:
                          English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:
                                                                    DATE
     PATENT NO.
                        KIND DATE
                                            APPLICATION NO.
                         ----
                                             -----
         003070683 A1 20030828 WO 2002-IN29 20020222
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
     WO 2003070683
             CO, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS,
             LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO,
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RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ,
             VN, YU, ZA, ZW
         RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY,
             KG, KZ, MD, RU, TJ, TM, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB,
             GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA,
              GN, GQ, GW, ML, MR, NE, SN, TD, TG
     AU 2002246303
                          A1
                                  20030909
                                              AU 2002-246303
                                                                       20020222
                                              WO 2002-IN29
                                                                   A 20020222
PRIORITY APPLN. INFO.:
                          CASREACT 139:197197
OTHER SOURCE(S):
     A process for preparing mineral acid addition salts of gabapentin (e.g.,
     gabapentin dihydrogen phosphate) comprises: (a) treating 1,
     1-cyclohexanediacetic acid monoamide with
     sodium hypobromite to effect a decarbonylation; (b) acidifying the
     reaction mass with a mineral acid (e.g., phosphoric acid) to a pH of about
     2; (c) extracting the acid addition salt with a ketone solvent (e.g., MEK); (d)
     evaporating the solvent; (e) dissolving the extract in an alc. solvent (e.g.,
     isopropanol); (f) filtering the undissolved material and evaporating the alc.
     solvent to obtain a syrupy residue; and (g) mixing the residue with
     non-polar organic solvents (e.g., toluene) to obtain mineral acid addition
     of gabapentin.
REFERENCE COUNT:
                          2
                                 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS
                                 RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT
L32 ANSWER 2 OF 2 HCAPLUS COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER:
                          2003:511296 HCAPLUS
DOCUMENT NUMBER:
                          139:85334
                          Preparation of benzyl cyclic amines such as
TITLE:
                          benzylpiperidine derivatives as serotonin reuptake
                          inhibitors
                          Kodo, Toru; Yagi, Hideki; Dan, Akihito; Masumoto,
INVENTOR(S):
                          Shuji; Kinomura, Naoya; Koyama, Koji
                          Sumitomo Pharmaceuticals Co., Ltd., Japan
PATENT ASSIGNEE(S):
SOURCE:
                          PCT Int. Appl., 186 pp.
                          CODEN: PIXXD2
DOCUMENT TYPE:
                          Patent
LANGUAGE:
                           Japanese
FAMILY ACC. NUM. COUNT:
PATENT INFORMATION:
                                  DATE APPLICATION NO.
                                                                     DATE
     PATENT NO.
                         KIND
                                              -----
                          ----
                                 -----
                                             WO 2002-JP13043
                                                                      20021212
                           A1
                                  20030703
     WO 2003053928
         W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN,
              CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH,
              GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KR, KZ, LC, LK, LR, LS,
              LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL,
              PT, RO, RU, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG,
              US, UZ, VC, VN, YU, ZA, ZM, ZW
         RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG
                                            AU 2002-366766
     AU 2002366766
                           A1
                                  20030709
                                                                       20021212
                                  20041013
                                              EP 2002-790747
                                                                       20021212
     EP 1466901
                           A1
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IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, SK

20050324

A1

US 2005065140

PRIORITY APPLN. INFO.:

AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT,

US 2004-498482 20040816

A 20011213

JP 2001-379598

JP 2001-399453 A 20011228 JP 2002-7140 A 20020116 WO 2002-JP13043 W 20021212

OTHER SOURCE(S):

MARPAT 139:85334

GΙ

$$Q^{1} = Q^{2} = X^{35}$$
 $X^{32}$ 
 $X^{31}$ 
 $X^{31}$ 
 $X^{32}$ 
 $X^{33}$ 
 $X^{34}$ 
 $X^{33}$ 
 $X^{34}$ 
 $X^{35}$ 
 $X^{34}$ 
 $X^{35}$ 
 $X^{36}$ 
 $X^{36}$ 

Disclosed is a serotonin reuptake inhibitor which contains as an active AB ingredient a cyclic amine represented by the formula (I) [wherein G = Q, -Z2-X20, Z3; R2 = H, halo, H0, each (un) substituted alkyl, alkoxy, or alkylthio; R3 = H, lower alkyl; Y = (un) substituted alkylene; n = 1, 2, 3; m= 0, 1,2,3; p = 1,2,3,4; wherein X10 = H, cycloalkyl, each (un) substituted alkyl, alkanoyl, alkanesulfonyl, alkylcarbamoyl, alkylsulfamoyl, alkoxycarbonyl, or amidino; X20 = HO, carbamoyloxy, each (un)substituted alkyl, NH2, alkoxy, or alkylcarbamoyloxy; Z2 = cycloalkane ring; Z3 = Q1, Q2; wherein X31 = a bond, CH2, CO; X32 = O, S, alkyl-(un)substituted NH; R6 = H, (un)substituted alkyl, cycloalkyl, aryl, or heteroaryl; X33 = a single bond, CH2, CO; X34 = a single bond, CH2; X35 = a single bond, CH2, O, S, alkyl-(un) substituted NH; provided that X34 and X35 are not simultaneously a single bond; R6 = H, alkyl; R8 = H, halo, alkyl, HO, (un) substituted alkoxy or alkylcarbamoyloxy], a prodrug thereof, or a pharmaceutically acceptable salt of any of these. The compds. I are selective serotonin reuptake inhibitors having an affinity for a serotonin 1A receptor. Thus, 55 mg triphosgene was added to a solution of 200 mg 3-[4-(2-bromo-5-methoxybenzyl)piperidin-1-yl]-1-cyclohexylaminopropan-2-ol and 0.083 mL Et3N in 5 m THF at room temperature and stirred for 6 h to give 100% 5-[[4-(2-bromo-5-methoxybenzyl)piperidin-1-yl]methyl]-3cyclohexyloxazolidin-2-one. 2-[[4-(2-Bromo-5-chlorobenzyl)piperidin-1yl]methyl]-1,2,3,4-tetrahydroquinoline dihydrochloride at 10-5 M increased by 74% the binding of [35S]GTPYS to CHO cell membrane expressing human 5-HTlA in the presence of 10  $\mu M$  serotonin (5-HT).

REFERENCE COUNT: 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS

RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

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(FILE 'HOME' ENTERED AT 11:48:54 ON 29 AUG 2007)
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     FILE 'HCAPLUS' ENTERED AT 11:50:33 ON 29 AUG 2007
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L1
     FILE 'REGISTRY' ENTERED AT 11:55:02 ON 29 AUG 2007
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     FILE 'HCAPLUS' ENTERED AT 11:55:03 ON 29 AUG 2007
                S E4
     FILE 'REGISTRY' ENTERED AT 11:56:31 ON 29 AUG 2007
L2
              1 S E4/CN
     FILE 'HCAPLUS' ENTERED AT 11:56:31 ON 29 AUG 2007
L3
              8 S L2
                E CYCLOHEXANEDIACETIC ACID ANHYDRIDE
         230564 S ANHYDRIDE
L4
              0 S L4 (3W) L3
L5
         230564 SEA ABB=ON PLU=ON ANHYDRIDE
L6
L7
          1633 S ABB=ON PLU=ON MONOAMIDE
         19338 S ABB=ON PLU=ON PRECIPITATION+PFT, OLD, NEW/CT
L8
              2 S ABB=ON PLU=ON ACIDIFICATION+PFT, NEW, OLD/CT
L9
L10
         214418 S ABB=ON PLU=ON AMMONIA
L11 .
              O S "HYDROCHLORIC ACID"+PFT, OLD, NEW/CT
                E HYDROCHLORIC ACID
L12
             12 S E5
L13
              O S ABB=ON PLU=ON "ACETIC ANHDRIDE"+PFT, NEW, OLD/CT
                E ACETIC ANHYDRIDE
         247054 S ACETIC
L14
L15
         26509 S L14 (1W) L4
        6805049 S ABB=ON PLU=ON SYNTH OR SYNTHE? OR PREPARTION OR PRODUC? OR PR
L16
        157279 S ABB=ON PLU=ON AMINATION+PFT, OLD, NEW, RT/CT
L17
                E US4024175/PN
              1 S E3
L18
                SELECT RN L18 1
     FILE 'REGISTRY' ENTERED AT 12:26:48 ON 29 AUG 2007
             11 S E1-E11
L19
              O S ABB=ON PLU=ON "1,1-CYCLOHEXANEDIACETIC ACID"+RTCS, NEW, OLD/C
L20
              O S ABB=ON PLU=ON GABAPENTINE+RTCS, NEW, OLD, PFT/CT
L21
L22
              7 S GABAPENTIN
              0 S L22 AND L2
L23
              0 S L22 AND L3
L24
     FILE 'HCAPLUS' ENTERED AT 12:40:47 ON 29 AUG 2007
           1956 S GABAPENTIN
L25
              0 S L25 AND L3
L26
             20 S L25 AND L6
L27
              4 S L27 AND L7
L28
             99 S 1,1-CYCLOHEXANEDIACETIC ACID
L29
              4 S L29 (3W) L6
L30
              3 S L30 NOT L28
L31
              2 S L29 AND L17 NOT L28
L32
=> 129 and 18
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08/29/2007 Page 30 of 44

L29 IS NOT A RECOGNIZED COMMAND

The previous command name entered was not recognized by the system. For a list of commands available to you in the current file, enter "HELP COMMANDS" at an arrow prompt (=>).

=> s 129 and 18

L33 2 L29 AND L8

=> d 133 1-2 ibib abs

L33 ANSWER 1 OF 2 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2006:15055 HCAPLUS

DOCUMENT NUMBER: 144:108021

TITLE: Process for the preparation of a gabapentin precursor INVENTOR(S): Villa, Marco; Paiocchi, Maurizio; Arrighi, Katiuscia;

Corcella, Francesco; Cannata, Vincenzo; Soriato,

Giorgio; Verzini, Massimo

PATENT ASSIGNEE(S): Zambon Group S.P.A., Italy

SOURCE: PCT Int. Appl., 18 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PA	rent 1	KINI	)	DATE					DATE								
						-											
WO	2006	0005	62		A1		2006	0105	ī	WO 2	005-1		20050622				
	W:	ΑE,	AG,	AL,	AM,	AT,	ΑU,	ΑZ,	BA,	BB,	BG,	BR,	BW,	BY,	BZ,	CA,	CH,
		CN,	CO,	CR,	CU,	CZ,	DE,	DK,	DM,	DZ,	EC,	EE,	EG,	ES,	FI,	GB,	GD,
		GE,	GH,	GM,	HR,	HU,	ID,	IL,	IN,	IS,	JP,	KΕ,	KG,	KM,	ΚP,	KR,	KZ,
		LC,	LK,	LR,	LS,	LT,	LU,	LV,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	MZ,	NA,
		NG,	NI,	NO,	NZ,	OM,	PG,	PH,	PL,	PT,	RO,	RU,	SC,	SD,	SE,	SG,	SK,
		SL,	SM,	SY,	TJ,	TM,	TN,	TR,	TT,	TZ,	UA,	UG,	US,	UZ,	VC,	VN,	YU,
		ZA,	ZM,	ZW													
	RW:	AT,	BE,	BG,	CH,	CY,	CZ,	DE,	DK,	EE,	ES,	FI,	FR,	GB,	GR,	HU,	ΙĖ,
		IS,	IT,	LT,	LU,	MC,	NL,	PL,	PT,	RO,	SE,	SI,	SK,	TR,	BF,	ВJ,	CF,
		CG,	CI,	CM,	GA,	GN,	GQ,	GW,	ML,	MR,	NE,	SN,	TD,	TG,	BW,	GH,	GM,
		KE,	LS,	MW,	MZ,	NA,	SD,	SL,	SZ,	TZ,	UG,	ZM,	ZW,	AM,	AZ,	BY,	KG,
		KZ,	MD,	RU,	TJ,	TM											
CA	2570	461			A1		2006	0105		CA 2	005-		20050622				
EP	1765	770			A1		2007	0328		EP 2	005-		2	0050	622		
	R:	AT,	BE,	BG,	CH,	CY,	CZ,	DE,	DK,	EE,	ES,	FI,	FR,	GB,	GR,	HU,	IE,
							MC,										
		HR,	LV,	MK,	YU												
IN	2006	CN04	728		Α		2007	0629		IN 2	006-	CN47	28		2	0061	222
PRIORIT												MI12				0040	624
									,	WO 2	005-	EP52	906	1	W 2	0050	622

OTHER SOURCE(S): CASREACT 144:108021

AB A process for the preparation of 1,1-cyclohexane acetic acid monoamide, an intermediate used in the preparation of gabapentin, comprises the basic hydrolysis reaction of  $\alpha,\alpha$ -diaminocarbonyl- $\beta,\beta$ -

pentamethylene glutarimide.

REFERENCE COUNT: 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L33 ANSWER 2 OF 2 HCAPLUS COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 2003:22835 HCAPLUS

DOCUMENT NUMBER: 138:73019

TITLE: Amidation process for the preparation of 1,

1-cvclohexanediacetic acid

monoamide from 1,1-cyclohexanediacetic anhydride and

aqueous ammonia

INVENTOR(S):

Oren, Jacob

PATENT ASSIGNEE(S):

Bromine Compounds Ltd., Israel

SOURCE:

PCT Int. Appl., 15 pp.

DOCUMENT TYPE:

CODEN: PIXXD2 Patent

LANGUAGE:

English

FAMILY ACC. NUM. COUNT:

PATENT INFORMATION:

	PAT	ENT 1	NO.			KIN	D :	DATE		APPLICATION NO. D								ATE		
	WO	2003002517			A1 20030109				,	WO 2	002-		20020617							
		W:	ΑE,	AG,	AL,	AM,	AT,	AU,	AZ,	BA,	BB,	BG,	BR,	BY,	BZ,	CA,	CH,	CN,		
			CO,	CR,	CU,	CZ,	DE,	DK,	DM,	DZ,	EC,	EE,	ES,	FI,	GB,	GD,	GE,	GH,		
			GM,	HR,	HU,	ID,	IL,	IN,	IS,	JP,	KE,	KG,	KP,	KR,	KZ,	LC,	LK,	LR,		
			LS,	LT,	LU,	LV,	MA,	MD,	MG,	MK,	MN,	MW,	MX,	MZ,	NO,	NZ,	OM,	PH,		
			PL,	PT,	RO,	RU,	SD,	SE,	SG,	SI,	SK,	SL,	ТJ,	TM,	TN,	TR,	TT,	TZ,		
			UA,	UG,	US,	UZ,	VN,	YU,	ZA,	ZM,	ZW									
		RW:	GH,	GM,	KE,	LS,	MW,	MZ,	SD,	SL,	SZ,	TZ,	UG,	ZM,	ZW,	AT,	BE,	CH,		
			CY,	DE,	DK,	ES,	FI,	FR,	GB,	GR,	ΙE,	IT,	LU,	MC,	NL,	PT,	SE,	TR,		
			BF,	ВJ,	CF,	CG,	CI,	CM,	GA,	GN,	GQ,	GW,	ML,	MR,	NE,	SN,	TD,	TG		
AU 2002311607					A1		2003	0303		AU 2002-311607					2	0020	617			
PRIOR	RITY	APP	LN.	INFO	.:						IL 2	001-	1440		A 20010628					
										,	WO 2	002-	IL47	3	1	W 2	0020	617		

OTHER SOURCE(S): CASREACT 138:73019

1,1-Cyclohexanediacetic acid

monoamide (CHDAAM), a gabapentin intermediate (no data), is prepared in high yield and selectivity by amination of 1,1-cyclohexanediacetic anhydride (CDAAn) with aqueous ammonia, followed by neutralization of the reaction mixture

with an acid (e.g., H2SO4) such that crude CHDAAM is precipitated, filtered, and

purified by crystallization from a solvent. The amination is carried out at <20° with aqueous ammonia having a concentration of 25-35% and in a molar ratio, relative to the CHDAAn, of 5-10, resp. The neutralization is carried out with an aqueous solution of H2SO4 having a concentration of 30-70%

continued until a slightly acid solution is obtained.

REFERENCE COUNT:

THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS 2 RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

=> s 129 and 19 not 128

0 L29 AND L9 NOT L28

=> file casreact

SINCE FILE COST IN U.S. DOLLARS TOTAL ENTRY SESSION 83.13 279.56 FULL ESTIMATED COST TOTAL DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS) SINCE FILE SESSION ENTRY -8.58 -8.58 CA SUBSCRIBER PRICE

FILE 'CASREACT' ENTERED AT 12:53:00 ON 29 AUG 2007 USE IS SUBJECT TO THE TERMS OF YOUR CUSTOMER AGREEMENT

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FILE CONTENT: 1840 - 25 Aug 2007 VOL 147 ISS 10

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Some CASREACT records are derived from the ZIC/VINITI database (1974-1999) provided by InfoChem, INPI data prior to 1986, and Biotransformations database compiled under the direction of Professor Dr. Klaus Kieslich.

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> s 1,1-cyclohexanediacetic acid 414776 1

414//6 1

414776 1

55 CYCLOHEXANEDIACETIC

218720 ACID

68281 ACIDS

231553 ACID

(ACID OR ACIDS)

L35 26 1,1-CYCLOHEXANEDIACETIC ACID

(1 (W) 1 (W) CYCLOHEXANEDIACETIC (W) ACID)

=> s amination

19881 AMINATION

168 AMINATIONS

L36 \ 19903 AMINATION

(AMINATION OR AMINATIONS)

=> s precipitation

235 PRECIPITATION

1 PRECIPITATIONS

L37 235 PRECIPITATION

(PRECIPITATION OR PRECIPITATIONS)

=> s monoamide

192 MONOAMIDE

77 MONOAMIDES

L38 243 MONOAMIDE

(MONOAMIDE OR MONOAMIDES)

=> s 1,1-cyclohexanediacetic acid anhydride

414776 1

414776 1

55 CYCLOHEXANEDIACETIC

218720 ACID

68281 ACIDS

231553 ACID (ACID OR ACIDS) 25163 ANHYDRIDE 4130 ANHYDRIDES 26192 ANHYDRIDE (ANHYDRIDE OR ANHYDRIDES) L39 1 1,1-CYCLOHEXANEDIACETIC ACID ANHYDRIDE (1 (W) 1 (W) CYCLOHEXANEDIACETIC (W) ACID (W) ANHYDRIDE) => s 135 and 136 2 L35 AND L36 => d 140 1-2 ibib abs L40 ANSWER 1 OF 2 CASREACT COPYRIGHT 2007 ACS on STN 138:73019 CASREACT ACCESSION NUMBER: Amidation process for the preparation of 1, TITLE: 1-cyclohexanediacetic acid monoamide from 1,1-cyclohexanediacetic anhydride and aqueous ammonia Oren, Jacob INVENTOR(S): Bromine Compounds Ltd., Israel PATENT ASSIGNEE(S): SOURCE: PCT Int. Appl., 15 pp. CODEN: PIXXD2 Patent DOCUMENT TYPE: English LANGUAGE: FAMILY ACC. NUM. COUNT: 1 PATENT INFORMATION: APPLICATION NO. DATE PATENT NO. KIND DATE \_\_\_\_\_\_ \_\_\_\_\_ ----\_\_\_\_\_ WO 2003002517 A1 20030109 WO 2002-IL473 20020617 W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG AU 2002-311607 20020617 AU 2002311607 A1 20030303 IL 2001-144066 20010628 PRIORITY APPLN. INFO.: 20020617 WO 2002-IL473 1.1-Cyclohexanediacetic acid AB monoamide (CHDAAM), a gabapentin intermediate (no data), is prepared in high yield and selectivity by amination of 1,1-cyclohexanediacetic anhydride (CDAAn) with aqueous ammonia, followed by neutralization of the reaction mixture with an acid (e.g., H2SO4) such that crude CHDAAM is precipitated, filtered, and purified by crystallization from a solvent. The amination is carried out at <20° with aqueous ammonia having a concentration of 25-35% and in a molar ratio, relative to the CHDAAn, of 5-10, resp. The neutralization is carried out with an aqueous solution of H2SO4 having a

RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS

of 30-70% and is continued until a slightly acid solution is obtained.

2

concentration

REFERENCE COUNT:

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L40 ANSWER 2 OF 2 CASREACT COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER:
                           54:80337 CASREACT
                           Compounds derived from \beta-substituted glutaric
TITLE:
                           acids: glutarimides, glutaramic acids,
                           1,5-pentanediols
                           Handley, G. J.; Nelson, E. R.; Somers, T. C.
AUTHOR(S):
CORPORATE SOURCE:
                           Nicholas Inst., Victoria
                           Australian Journal of Chemistry (1960), 13, 127-44
SOURCE:
                           CODEN: AJCHAS; ISSN: 0004-9425
DOCUMENT TYPE:
                           Journal
                           Unavailable
LANGUAGE:
     \alpha,\alpha'\text{-Dicyano-}\beta,\beta\text{-dialkylglutarimides} (I) were
     obtained by the Guareschi synthesis (CA 14, 172) the scope of which was
     discussed. \beta, \beta-Dialkylglutaric acids (II) were prepared by
     dissolving 0.5 mole I in 250 ml. warm, concentrated H2SO4, adding 250 ml. H2O
     slowly with shaking and refluxing the mixture until no solid remained (8
     hrs.). The oily layer which separated solidified on cooling and was collected
     and dissolved in ether. The solution was washed with H2O, extracted with
saturated
     NaHCO3 solution, the extract acidified and extracted with Et20. Evaporation
of the Et20
     left a residue which could be recrystd. from ligroine-benzene. Diethyl
     β,β-dialkylglutarates (III) were prepared from II by refluxing
     with EtOH-H2SO4. β,β-Dialkylglutarimides (IV) were prepared
     directly from 0.5 mole II by heating 2-3 hrs. with 1 mole urea in an oil
     bath at 170-90° and recrystg. from H2O or EtOH.
     \beta, \beta-Dialkyl-N-substituted-glutarimides (V) were obtained from
     0.1 mole anhydride of II (prepared by refluxing II with Ac20) and 0.1 mole
     appropriate primary amine by mixing and then heating in an oil bath at
     170-90° for 2-3 hrs. The following I and their products were
     prepared \alpha, \alpha'-Dicyano-\beta, \beta-dimethylglutarimide (69%
     yield), m. 214-15°, gave \beta,\beta-dimethylglutaric acid (72%),
     m. 102-3°; diethyl ester (67%), b2 93°, n2D 1.4290; imide
      (80%), m. 144°; N-Me imide (81%), m. 56-8°.
     \alpha, \alpha'-Dicyano-\beta-ethyl-\beta-methylglutarimide (73%), m.
     192-3°, gave \beta-ethyl-\beta-methylglutaric acid (89%), m.
     86-7°; diethyl ester (76%) b3 110°, n21D 1.4343; imide
      (71%) m. 126-7°; N-Me imide (61%), b7 126-30°, n22D 1.4743;
     N-Et imide (60%), b6 110-12°, n22D 1.4709; N-Ph imide (65%), m.
      128°; N-(p-MeC6H4) imide (74%), m. 150-1°, N-PhCH2 imide
      (69%), b1 160°, n22D 1.5294. \alpha,\alpha'-Dicyano-\beta-
     methyl-\beta-propylglutarimide (79%), m. 202-3°, gave
      \beta-methyl-\beta-propylglutaric acid (86%), m. 89-91°; diethyl
      ester (70%) b3.5 114-15°, n21D 1.4359; imide (46%) m.
      119-20°. \alpha, \alpha'-Dicyano-\beta-methyl-\beta-
      isopropylqlutarimide (13%), m. 234-6°, gave \beta-methyl-\beta-
      isopropylglutaric acid (90%), m. 100°; imide (90%) m. 165-6°; N-Me imide (75%) b25 165-70°, n22D 1.4812.
      \alpha, \alpha'-Dicyano-\beta-butyl-\beta-methylglutarimide (77%), m.
      174-5°, gave \beta-butyl-\beta-methylglutaric acid (85%), m.
      63-5°; diethyl ester (60%) b13 168°, n28D 1.4339; imide
      (63%) m. 115-15.5°; N-Me imide (67%) b7 140°, n20D 1.4762;
      N-Et imide (63%) b6 136°, n20D 1.4712. α,α'-Dicyano-
      \beta-isobutyl-\beta-methylglutarimide (39%), m. 237-8°, gave
      \beta-isobutyl-\beta-methylglutaric acid (74%), m. 66-8°; imide
      (90%) m. 103-4°. \alpha,\alpha'-Dicyano-\beta-methyl-\beta-
      pentylglutarimide (65%), m. 166-7°, gave \beta-methyl-\beta-
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pentylglutaric acid (91%, including 1/5 as imide), m. 73-4°;
     diethyl ester (70%) b14 170°, n21D 1.4392; imide (74%) m.
     113-14°; N-Me imide (56%) b4 132°, m. 43-4°; N-Et
     imide (61%) b5 142°, n21D 1.4708. \alpha,\alpha'-Dicyano-\beta-
     methyl-β-isopentylglutarimide (62%), m. 196-7°, gave
     \beta-methyl-\beta-isopentylglutaric acid (91%), m. 70-73°; imide
     (78%) m. 120-1°. \alpha, \alpha'-Dicyano-\beta-hexyl-\beta-
     methylglutarimide (50%) m. 152-3°, gave \beta-hexyl-\beta-
     methylglutaric acid (76%, including 1/3 as imide), m. 66-7°;
     diethyl ester (73%) b4 164°, n21D 1.4414; imide (60%) m.
     99.5-100.5°; N-Me imide (49%) b7 162°, n21D 1.4745; N-Et
     imide (57%) b6 160°, n21D 1.4717. \alpha,\alpha'-Dicyano-\beta-
     isohexyl-\beta-methylglutarimide (64%), m. 161-6°, gave
     \beta-isohexyl-\beta-methylglutaric acid (92%, including 1/3 as imide),
     m. 79-80°; imide (90%) m. 107-8.5°. \alpha,\alpha'-
     Dicyano-β-heptyl-β-methylglutarimide (58%), m.
     147.5-9.0°, gave on hydrolysis only \beta-heptyl-\beta-
     methylglutarimide (90%), m. 95-6° (cf. Birch and Robinson, CA 37,
     6033). \alpha, \alpha'-Dicyano-\beta-methyl-\beta-nonylglutarimide
     (80%), m. 136°, gave \beta-methyl-\beta-nonylglutaric acid (78%,
     including 1/2 as imide), m. 44-7°; imide (85%) m. 100°.
     \alpha,\alpha'\text{-Dicyano-}\beta\text{-methyl-}\beta\text{-phenylglutarimide} (2%, and
     27% by the method of McElvain and Clemens, CA 53, 3215c), m. 275°,
     gave on hydrolysis with 50-55% weight/weight H2SO4 under reflux for 24 hrs.
     β-methyl-β-phenylglutaric acid (82%), m. 140-2°; imide
     (90%) m. 156-7°. \alpha, \alpha'-Dicyano-\beta-benzyl-\beta-
     methylglutarimide (76%), m. 249-50°, decomposed under the hydrolysis
     conditions (Kon and Stevenson, CA 15, 1279). \alpha,\alpha'-Dicyano-
     \beta, \beta-diethylglutarimide (35%), m. 204-6°, gave
     \beta, \beta-diethylglutaric acid (81%), m. 108°; imide (74%) m.
     146-7°. \alpha, \alpha'-Dicyano-\beta, \beta-dipropylglutarimide
     (9%), m. 215-16°, gave \beta, \beta-dipropylglutaric acid (70%),
     m. 117-18°; imide (71%) m. 125°. \alpha, \dot{\alpha}'-Dicyano-
     \beta\text{-spiro}(\text{cyclopentane}) glutarimide (17%), m. 179-80°, gave
     \beta-spiro(cyclopentane)glutaric acid (80%), m. 177-8°; imide
     (56%), m. 153°. \alpha,\alpha'-Dicyano-\beta-
     spiro(cyclohexane) glutarimide (74%), m. 208-10°, gave
     \beta-spirocyclohexane glutaric acid (93%), m. 182-3°; diethyl
     ester (78%) b3.5 138-40°, n25D 1.4573; imide (80%) m.
     169-70°; N-Me imide (67%) m. 69-70°. \alpha,\alpha'-
     Dicyano-\beta-spiro(\alpha-methylcyclohexane)glutarimide (16%), m.
     243° (decomposition), gave \beta-spiro(\alpha-
     methylcyclohexane)glutaric acid (82%), m. 143-5°; imide (60%) m.
     122-4°. \alpha, \alpha'-Dicyano-\beta-
     spiro(cycloheptane)glutarimide (8%), m. 205-6°, gave
     β-spiro(cycloheptane) glutaric acid (62%), m. 155-6°; imide
     (71%) m. 177-8°. No imide was obtained in the Guareschi synthesis
     from MeCH(OH)Ac, AcCH2CO2Et, Et2N(CH2)2Ac, EtCO(CH2)4Me, EtCOPh,
     iso-Bu2CO, or Ph2CO. A series of 2,2-dialkyl-1,1,3,3-tetracarboxy propane
     diimides (VI) were prepared by dissolving 5 g. of the appropriate I in 20
     ml. 60% H2SO4. The solution was heated several min. until a white solid
     appeared and for a further 1 min., then cooled, the precipitate collected,
washed
     with H2O and recrystd. from H2O. Thus were prepared 2,2-dimethyl-1,1,3,3-
     tetracarboxypropane diimide (45% yield), m. above 360°,
     2-ethyl-2-methyl-1,1,3,3-tetracarboxypropane diimide (38%), m.
     329-31°, 2-methyl-2-propyl-1,1,3,3-tetracarboxypropane diimide
     (85%), m. 278-80°, 2-butyl-2-methyl-1,1,3,3-tetracarboxypropane diimide (75%), m. 278-80°, 2-methyl-2-pentyl-1,1,3,3-
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tetracarboxypropane diimide (70%), m. 237-8°, 2-hexyl-2-methyl-
     1,1,3,3-tetracarboxypropane diimide (55%), m. 214°, and
     2-spiro(cyclohexane)-1,1,3,3-tetracarboxypropane diimide (65%), m. above
     370°. Hydrolysis of I with cold, concentrated H2SO4 for 24 hrs. (Thorpe
     and Wood, CA 8, 490) gave mixts. of VI and the corresponding
     \alpha, \alpha'-dicarboxamido-\beta, \beta-dialkylqlutarimides, except
     with \alpha, \alpha'-dicyano-\beta, \beta-dimethylglutarimide which gave
     only \alpha, \alpha'-dicarboxamido-\beta, \beta-dimethylglutarimide
     (70%), m. above 330° (H2O), and with \alpha,\alpha'-dicyano-
     \beta-ethyl-\beta-methylglutarimide which gave only \alpha, \alpha'-
     dicarboxamido-\beta-ethyl-\beta-methylglutarimide (75%), m. 277°
     (decomposition). A series of \beta, \beta-dialkylglutaramic acids were prepared
     by refluxing the 0.1 mole appropriate IV with 0.1 mole NaOH in 35 ml. H2O
     for 30 min. The solution was cooled, filtered, acidified with concentrated
HCl,
     the precipitated oil solidified by rubbing, and the solid recrystd. from H2O.
     Thus were prepared \beta-ethyl-\beta-methylglutaramic acid (85% yield), m.
     84-5°, \beta-methyl-\beta-propylglutaramic acid (88%), m.
     98-8°, \beta-methyl-\beta-isopropylglutaramic acid (85%), m.
     118°, \beta-butyl-\beta-methylglutaramic acid (93%), m.
     88-91°, \beta-isobutyl-\beta-methylglutaramic acid (90%), m.
     96°, \beta-methyl-\beta-pentylglutaramic acid (95%), m.
     86-8°, β-hexyl-β-methylglutaramic acid (82%), m.
     63-5°, β,β-diethylglutaramic acid (90%), m. 130°,
     β-spiro(cyclopentane) glutaramic acid (85%), m. 110°, and
     \beta-spiro(cyclohexyl)glutaramic acid (80%), m. 141-5°. The
     series of 3,3-dialkyl-1,5-pentanediols (VII) prepared by reduction of the
     appropriate III with LiAlH4 in Et20 included: 3,3-dimethyl-1,5-pentanediol
     (50% yield), b3 131°, n26D 1.4510; 3-ethyl-3-methyl-1,5-pentanediol
     (85%), b10 160-2°, n23D 1.4640; 3-methyl-3-propyl-1,5-pentanediol
     (75%), b3 144, n26D 1.4618; 3-butyl-3-methyl-1,5-pentanediol (83%), b0.5
     134°, n26D 1.4629; 3-methyl-3-pentyl-1,5-pentanediol (80%), bl
     168-9°, n23D 1.4640; 3-hexyl-3-methyl-1,5-pentanediol (92%) b1,
     182-4°, n25D 1.4631; 3-spiro(cyclohexane)-1,5-pentanediol (62%), b2
     166°, n25D 1.4930. A series of \beta-alkylglutaric acids was
     prepared by condensation of the appropriate aldehydes with NCCH2CONH2 (cf.
     Day and Thorpe, CA 15, 1134) and hydrolysis of the resulting
     \alpha, \alpha'-dicyano-\beta-alkylglutarimides (VIII) as above for I.
     Thus were prepared: \beta-methylglutaric acid (71% yield from VIII), m.
     84-5° [imide (58%) m. 142-3°; N-phenethyl imide (61%), m.
     100-1°]; \beta-ethylglutaric acid (74%), m. 67-9° [imide
     (53%), m. 84-5°]; \beta-propylglutaric acid (70%), m.
     48-50° [imide (70%) m. 113-13.5°]; \beta-isobutylglutaric
     acid (50%), m. 47-8° [imide (70%) m. 138-8.5°];
     β-hexylglutaric acid (62%), m. 36-8° [imide (72%) m.
     112-13°]. \beta-Phenylglutaric acid gave the imide (75%), m.
     173-4°, N-Me imide (62%), m. 141-2°, and N-Et imide (79%),
     m. 94°. Hypnotic, convutsant, analeptic, and barbiturate-
     antagonistic effects of IV, and sedative and hypnotic effects of VII are
     discussed. The other series had no appreciable pharmacol. activity.
=> s acidification
           3187 ACIDIFICATION
              1 ACIDIFICATIONS
           3188 ACIDIFICATION
                   (ACIDIFICATION OR ACIDIFICATIONS)
=> d his
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L41

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L4
L5
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L16
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L36
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=> s 138 and 135
        6 L38 AND L35
L42
=> s 142 and 137
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            2 L42 AND L37
=> d 143 1-2 ibib abs
L43 ANSWER 1 OF 2 CASREACT COPYRIGHT 2007 ACS on STN
                       144:108021 CASREACT
ACCESSION NUMBER:
                       Process for the preparation of a gabapentin precursor
TITLE:
                       Villa, Marco; Paiocchi, Maurizio; Arrighi, Katiuscia;
INVENTOR(S):
                       Corcella, Francesco; Cannata, Vincenzo; Soriato,
                       Giorgio; Verzini, Massimo
                       Zambon Group S.P.A., Italy
PATENT ASSIGNEE(S):
                       PCT Int. Appl., 18 pp.
SOURCE:
                       CODEN: PIXXD2
DOCUMENT TYPE:
                       Patent
                       English
LANGUAGE:
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:
                                 APPLICATION NO. DATE
    PATENT NO.
                 KIND DATĒ
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    WO 2006000562 A1 20060105 WO 2005-EP52906 20050622
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            CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG, BW, GH, GM,
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EP 2005-776192 20050622
                    A1 20060105
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                    A1 20070328
     EP 1765770
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                   A 20070629
                                        IN 2006-CN4728
                                                         20061222
     IN 2006CN04728
PRIORITY APPLN. INFO.:
                                         IT 2004-MI1271
                                                         20040624
                                        WO 2005-EP52906 20050622
```

AB A process for the preparation of 1,1-cyclohexane acetic acid monoamide , an intermediate used in the preparation of gabapentin, comprises the basic hydrolysis reaction of  $\alpha,\alpha$ -diaminocarbonyl- $\beta,\beta$ -

pentamethylene glutarimide.

REFERENCE COUNT: 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L43 ANSWER 2 OF 2 CASREACT COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 138:73019 CASREACT

TITLE: Amidation process for the preparation of 1,

1-cyclohexanediacetic acid

monoamide from 1,1-cyclohexanediacetic

anhydride and aqueous ammonia

INVENTOR(S): Oren, Jacob

PATENT ASSIGNEE(S): Bromine Compounds Ltd., Israel

SOURCE: PCT Int. Appl., 15 pp.

CODEN: PIXXD2

DOCUMENT TYPE:

LANGUAGE:

Patent English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

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APPLICATION NO. DATE
     PATENT NO. KIND DATE
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     WO 2003002517 A1 20030109 WO 2002-IL473 20020617
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               PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ,
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RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG
     AU 2002311607
                        A1 20030303
                                                  AU 2002-311607 20020617
PRIORITY APPLN. INFO.:
                                                   IL 2001-144066
                                                                       20010628
                                                   WO 2002-IL473 20020617
```

AB 1,1-Cyclohexanediacetic acid

monoamide (CHDAAM), a gabapentin intermediate (no data), is prepared in high yield and selectivity by amination of 1,1-cyclohexanediacetic anhydride (CDAAn) with aqueous ammonia, followed by neutralization of the reaction mixture with an acid (e.g., H2SO4) such that crude CHDAAM is precipitated,

filtered, and purified by crystallization from a solvent. The amination is carried out at <20° with aqueous ammonia having a concentration of 25-35% and in a molar ratio, relative to the CHDAAn, of 5-10, resp. The neutralization is carried out with an aqueous solution of H2SO4 having a concentration

of 30-70% and is continued until a slightly acid solution is obtained.

REFERENCE COUNT: 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

=> s ammonia

8351 AMMONIA 3 AMMONIAS 8354 AMMONIA

(AMMONIA OR AMMONIAS)

=> s 144 and 129 414776 1

414776 1

55 CYCLOHEXANEDIACETIC

218720 ACID

68281 ACIDS

231553 ACID

(ACID OR ACIDS)

26 1,1-CYCLOHEXANEDIACETIC ACID

(1 (W) 1 (W) CYCLOHEXANEDIACETIC (W) ACID)

L45 3 L44 AND L29

=> s 145 not 128

74 GABAPENTIN

25163 ANHYDRIDE

4130 ANHYDRIDES

26192 ANHYDRIDE

(ANHYDRIDE OR ANHYDRIDES)

192 MONOAMIDE

77 MONOAMIDES

243 MONOAMIDE

(MONOAMIDE OR MONOAMIDES)

L46 1 L45 NOT L28

=> d l46 ibib abs

L46 ANSWER 1 OF 1 CASREACT COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER: 136:19880 CASREACT

TITLE: Preparation of 1-(2-amino-2-oxoethyl)cyclohexaneacetic

acid

INVENTOR(S): Tang, Miaorong; Fan, Weirong; Liu, Tianchun; Zhang,

Xiaobo

PATENT ASSIGNEE(S): Hangzhou Shouxin Fine Chemical Co., Ltd., Peop. Rep.

China

SOURCE: Faming Zhuanli Shenqing Gongkai Shuomingshu, 5 pp.

CODEN: CNXXEV

DOCUMENT TYPE: Patent LANGUAGE: Chinese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
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CN 1297885	A	20010606	CN 2000-128111	20001201
CN 1109017	B	20030521		

PRIORITY APPLN. INFO.: CN 2000-128111 20001201

AB 1-(2-Amino-2-oxoethyl)cyclohexaneacetic acid is synthesized by condensing cyclohexanone with Et cyanoacetate in ethanol under bubbling NH3 for 18-26 h, stirring at 0° for 18-26 h and at 25° for 100-130 h to obtain  $\alpha,\alpha$ -dicyano-1,1-cyclohexanediacetimide ammonium salt, hydrolyzing with H2SO4 solution at 200° for 30 min to obtain 1,1-cyclohexanediacetic acid, dehydrating with acetic anhydride to obtain 1,1-cyclohexanediacetic anhydride, aminolyzing with NH3 or NH4OH at 30-110° for 3-8 h, and recrystg. with ethanol.

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(FILE 'HOME' ENTERED AT 11:48:54 ON 29 AUG 2007)

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     FILE 'HCAPLUS' ENTERED AT 11:50:33 ON 29 AUG 2007
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     FILE 'REGISTRY' ENTERED AT 11:56:31 ON 29 AUG 2007
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L5
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L6
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L8
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L9
L10
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L12
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L14
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L15
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L29
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08/29/2007 Page 42 of 44

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L37	235	S	PRECIPITATION
L38	243	S	MONOAMIDE
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L41	3188	S	ACIDIFICATION
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L43	2	S	L42 AND L37
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L4	8	((ANDREA) near2 (NICOLI)).INV.	US-PGPUB; USPAT; USOCR	OR	ON	2007/08/29 15:32
L5	21	((MAURIZIO) near2 (PAIOCCHI)). INV.	US-PGPUB; USPAT; USOCR	OR	ON	2007/08/29 15:33
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